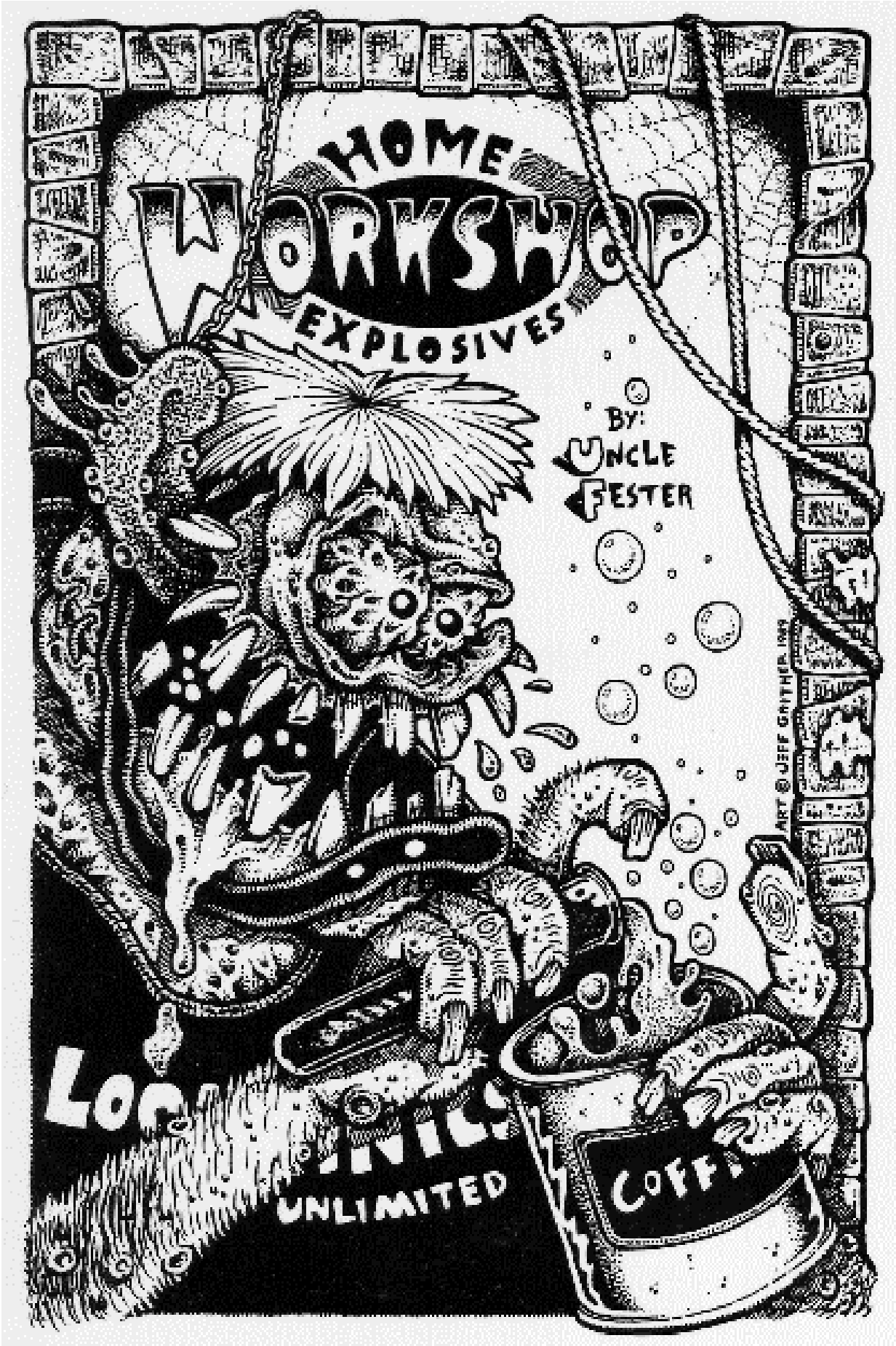
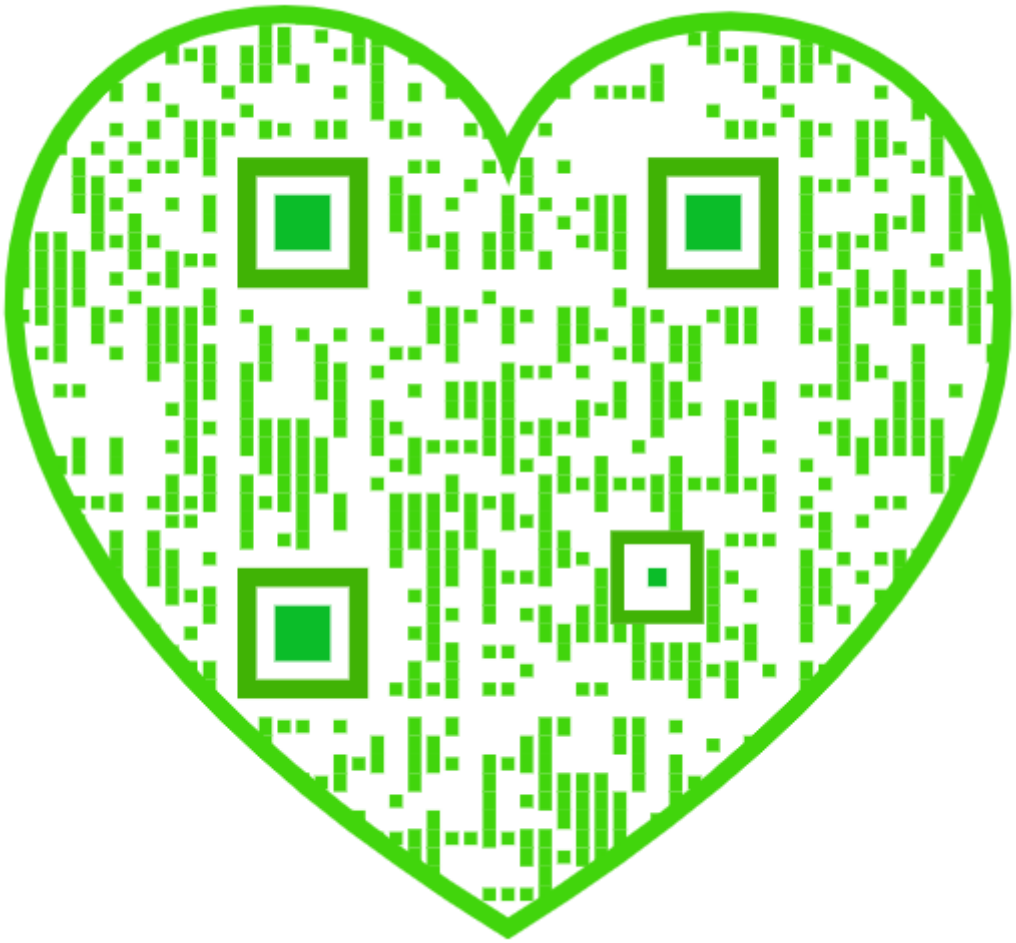

HOME
WORKSHOP
EXPLOSIVES
BY:
UNCLE
FESTER
ART © JEFF GRITHER 1993
UNLIMITED
COFF



Home Workshop Explosives
by Uncle Fester
Published by Loompanics Unlimited 1990
Digital transformation by Swedish Infomania 1996

PREFACE

To many people, it seems only natural that their Big Brother deems them unfit or untrustworthy to possess whole classes of substances. After all, they are only lowly serfs, and are not intelligent enough or stable enough to handle explosives or the modem scourge of "killer dope." If this is the way you think, don't bother to continue reading this book, as it will only upset you.
If this stunted mode of thinking is as repugnant to you as it is to me, the next question to be dealt with is: How, in an unfree, heavily surveilled country, where the citizens are trained from infancy to be informers for the state, does one obtain explosive materials without being arrested or killed? For the vast majority of us who do not have access to the "legitimate" explosives supply pipeline, there are only two possible routes to take to obtain explosives. The first and most obvious possibility is to steal the explosive from some outfit that has a large, unguarded legitimately obtained supply, most likely a quarry. This is not such a good method for reasons I will outline. First of all, a theft of explosives never fails to catch the attention of the authorities in a particular area. There is just something about explosives that makes such people nervous, especially if there is a chance they have fallen into the "wrong hands." The intense scrutiny that is likely to follow such a theft is almost certainly unwelcome, and is bound to take away most of the pleasure and fun of owning explosives. Secondly, the performance of heisted explosives is most probably going to be disappointing. This is because the most easily stolen dynamites, those ripped off from quarries, are in all probability going to be low-powered rubbish. This kind of trash works fine for blasting loose rock, but is next to useless for demolition purposes. Finally, ever since the days of the Weathermen bombing campaign of the early '70's, all dynamites manufactured in the US have been laced with unique mixtures of trace elements to identify the manufacturer and batch number of the dynamite, even after it has been exploded. (This is done by scraping up some of the residue and analysing it.) As you can imagine, this identification system makes it much easier to prove whose stick of dynamite did what, or from whence a particular stick of dynamite came. For the above reasons, a second alternative will be covered in detail in this book. That alternative is the home manufacture of explosive mixtures suitable for demolition. The emphasis in this book will be to cover the techniques and equipment suitable for home manufacture of a limited number of very powerful explosives using as starting materials easily obtained chemicals. Here, the long suit is going to be detail of presentation, making the reader aware of the little things that can go wrong in production of explosives and how to avoid these little pitfalls. It is my opinion that this course of action is much preferable to the presentation given in other so called "explosives" books, where the authors attempt to cover up their own lack of detailed knowledge of the field by going through superficial litanies of a plethora of formulas for making various explosives. I find such a treatment of the topic insulting.

The material in this book is largely the result of numerous experiments I did in the field in my dorm room back when I was in college. I know from experience that these procedures will work for anybody so long as reasonable care is taken. You can believe it because I've been there, and wouldn't tell you so if it were not true.

Have fun!
Uncle Fester

THINGS THAT GO BOOM IN THE NIGHT

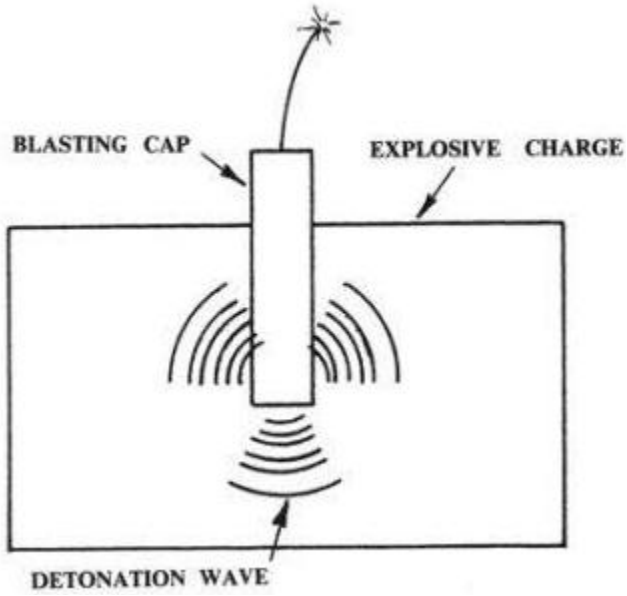
The first step towards gaining an understanding of explosives is to understand how they work. From this piece of knowledge, an appreciation for inany of the finer points of explosive technology will naturally flow.

An explosive is a substance or a mixture (usually a solid or a jelly in form) that is unstable. This unstable substance undergoes a very rapid chemical reaction after being set off in the proper manner. The result is that the originally rather small amount of explosive is almost instantly converted into a very large amount of hot gases. These hot gases are at first trapped into the amount of space that the explosive charge originally occupied, but they expand very rapidly and violently. The result is the familiar sight of an explosive fireball and its accompanying shock wave.

As we all know, not all explosives are created equal. Some explode so fiercely that they destroy everything within range, while others are useful for fireworks, but not much more. Why the wide variance in power? The answer to that lies in the three main factors contributing to an explosives performance.

The first and generally the most important factor in determining the performance of an explosive is its detonation rate. To understand what is meant by the term detonation rate, see the drawing below.

The drawing shows a charge of explosive in which a blasting cap has just gone off, triggering a detonation. Pay close attention to the detonation wave spreading outward in all directions from its source at the blasting cap. On the outward side of this front, the explosive is unchanged, but on the inward side, it has been detonated and converted to hot gases. The rate that this detonation wave travels through the explosive is called the detonation rate. This number is usually expressed in the form of meters travelled per second (m/s). It is not bard to imagine why a more powerful explosive will have the higher detonation rate. The more instantly the mass of explosive is converted into hot gases, the less chance these gases will have to fritter away without doing any damage. Typical detonation rates range from over 8000 m/s for cyclonite (RDX) down to a couple thousand for black powder.



Just as different explosives have different detonation rates, the same explosive under varying conditions will have a range of detonation rates. For a solid explosive such as RDX or PETN, this rate depends largely upon how dense the mass of explosive is. For example, if the charge consists of finely crystalline material, its detonation rate will be quite a bit less than if it has been melted together into one solid rock. This is because the finely powdered material is less dense than the rock form. Those readers familiar with cocaine will grasp this immediately because cocaine, like these solid explosives, fluffs up quite a bit in the process of being powdered up from rock form.

A liquid explosive like nitro is always going to have the same density, so it does not exhibit this effect. This, however, does not mean that the liquid explosive will always have the same detonation rate. When the pure liquid explosive is mixed with binders to make a safer explosive, its detonation rate goes down quite a bit. In the case of nitro, when it is mixed with binders to make dynamite, its detonation rate can be cut in half. The solid explosives like PETN or RDX also show this effect when they are mixed with binders to make plastique. The general rule is that the pure substance will be a good deal more powerful (although less safe and convenient to handle) than the resulting plastique or dynamite.

The second factor determining the performance of an explosive is how much hot gas a given amount of explosive will be converted to when it detonates. This factor is just as important as the detonation rate in determining how violent the resulting explosion will be. Unlike the detonation rate, this factor is not influenced by the density of the explosive or what binders it may be mixed with. One gram of black powder will only produce 270 c.c. of gas (volume of gas adjusted back to room temperature and normal pressure by use of the ideal gas law) while the same amount of RDX will make over 1000 c.c. of gas. Since it is this conversion of the explosive to gases that makes the blast, the extreme importance of this factor is easy to understand.

The final main factor determining how powerful an explosive is, is how much heat is produced in the blast (i.e., how hot the resulting gases are). This is because gases expand when they are heated, so the same amount of gas when reduced to standard pressure and room temperature can be a good deal greater when hot. Black powder produces about 700 calories of heat per gram, while nitro and PETN make in the neighbourhood of 1500 calories per gram.

If one takes these three factors together, you can get a good measure of the relative power of the explosives. A convenient way to do this is just to multiply the three factors together to get a number with which to compare the various explosives. A list of explosives compiled in this manner according to power would have RDX, PETN, nitro and a few of its close cousins crowded at the top, and then a middle group of which TNT is a typical example, and then a lower group including black powder and mercury fulminate.

Most members of this lower group do not detonate like the high explosives do. They explode in a different manner called deflagration. Deflagration can be most closely compared to a rapid burning. The outer surfaces of the individual particles catch fire and burn their way into the centres of the particles. With these explosives, maximum power is obtained by very finely granulating the material. This is in contrast to the high explosives where the greatest power is obtained when the pure material, if it is a solid, is in the form of a large solid rock. These lower explosives find most of their use in blasting caps to provide the initial shock that sets off the high explosive and as propellants or primers for bullets.

As you probably already know, the high explosives require a fairly violent blow to set them off. The forcefulness of the shock needed to cause detonation varies from explosive to explosive. TNT is very hard to set off, while on the other extreme, nitro and PETN are pretty easy to bring to detonation. To meet these varying needs, a range of blasting caps with increasing power, labelled numbers 1 through 8 are available. With almost all the explosives detailed in this book, I will note which number blasting cap is required to set it off. A larger blasting cap than needed to set the material off is generally not recommended, but may sometimes be advantageous by causing a condition called "overdriving" in which a more powerful blast is obtained.

Some of the high explosives covered in this work can also be set off by exposure to heat or a strong electric shock. Where this is applicable, it will be noted, along with some suggestions for taking advantage of this property.

Finally, I must warn the reader that only the most powerful of explosives are covered in this book. A small amount of them could easily make the careless experimenter a memory. These materials become most dangerous when the handler ceases to be afraid of them! For this reason they are not recommended as an adjunct to drunken revelry or other tomfoolery. Be warned!

THE NITRIC ESTERS

Since all of the compounds dealt with in this book, with the exception of RDX (cyclonite) belong to a class of chemicals called nitric esters, this discussion will begin by explaining exactly what are nitric esters, how they are made, what precautions can be followed by the home experimenter to get maximum yields of product, and some of the pitfalls likely to ensnare the unwary experimenter during the stages of production, purification and use of this most powerful class of explosives.

The layman who goes to the library and brings home an armful of good explosive books is sure to see the term "nitric ester" used repeatedly, with the predictable response of "Huh?" being the result. Since this class of explosives is so important, and forms the heart of Home Workshop Explosives, this term must be explained. An ester is a member of a family of related chemicals, all of whom can be made by reacting together an alcohol and an acid (or its derivatives, for those organic chemistry purists out there). The acid and the alcohol link together by splitting off a water molecule, and form the new compound, an ester. A nitric ester is an ester formed when the acid is nitric acid. A very large variety of nitric esters can be made from nitric acid, just by using a variety of alcohols. It is likely that all of them are explosive. The simplest nitric ester, methyl nitrate, is made by reacting together methyl alcohol (wood alcohol easily found at the local hardware store) and nitric acid. The following shows how these two react together to form an ester. All nitric esters in this book are formed by the same mechanism:

$$CH_3OH + HNO_3 => CH_3ONO_2 + H_2O$$

Methyl Alcohol + Nitric Acid => Methyl Nitrate

At the end of this chapter, the details of how to prepare methyl nitrate will be covered to illustrate the points made in this chapter.

Similarly, ethyl nitrate is made from ethyl alcohol (grain alcohol, the drinking variety) and nitric acid. The infamous nitroglycerin is the product of nitric acid and glycerin (glycerol). This same analogy can be carried throughout the entire class of chemicals called the nitric esters.

For the person interested in producing these chemicals, a main area of concern is naturally going to be how to get the maximum yield product from each batch. For the industrial-scale manufacturer, the emphasis may shift to how to get the cheapest product in the least amount of time. To answer these questions, the process of forming the nitric esters must be looked at more closely.

The process of forming a nitric ester belongs to a class of reactions called equilibrium reactions. What is meant by this is that all of the starting materials do not automatically and rapidly get converted into products. A good percentage of the starting materials (in the case of nitro, glycerin and nitric acid) are likely to just lay around in the mixture and refuse to react together.

This frustrating course of events can be avoided for the most part by a knowledge of what's going on, and using strategy to shape events to our liking.

Just how much of the starting materials are going to be turned into products can be figured out using a little algebra and what is called the equilibrium equation. Let's take the case of nitro production since nitro is such an effective explosive, and making it is required for the creation of gelatin dynamite. This is the reaction that forms nitro:

1 Glycerin + 3 Nitric Acid => 1 Nitroglycerin + 3 Water

Here we see that one molecule of glycerin and three molecules of nitric acid are required to make one molecule of nitroglycerin and three molecules of water. The equilibrium formula for this reaction looks like this:

$$\text{Constant} = \frac{(\text{nitro})*(\text{water})^3}{(\text{glycerin})*(\text{nitric acid})^3}$$

Here the constant is in the neighbourhood of 50, but depends upon the temperature that the reaction is being done at. What this equation means is that the reaction goes forward making nitro until the concentration of nitro in the mixture times the concentration of water to the third power, divided by the concentration of nitric acid times the concentration of glycerin to the third power reaches the value of the constant.

Now for the point of all this. In order for good yields of product to be obtained, it is crucial that there be as little water as possible in the mixture, and that the concentration of nitric acid be as high as possible. With a little bit of high school level algebra, you can easily prove to yourself that a little bit of water in the mix, since its concentration is raised to the third power, will wreak havoc on the amount of nitro allowed to be produced before the constant number is reached.

There are several methods for keeping the amount of water in the mix to a minimum. First of all, glycerin used in the reaction should not have any water in it. This can present a problem because the best source of glycerin is the local pharmacy, and the bottle it comes in is not likely to say how much water it contains. I have found a wide variety of quality in drug store glycerin. Some, like the Gull Pharmacy brand which was my favourite, was virtually pure glycerin

with no added water. Other rubbish brands which will not be named here had unacceptably high amounts of water in them. How to deal with this will be covered in more detail in the nitro section, but let me say right here that the more runny the glycerin is, the more water it contains.

Another very common method to keep the amount of water in the mix to a minimum is to add a substance which will soak up water in the reaction mixture. The most favoured chemical for this purpose is concentrated sulfuric acid. This material has a great thirst for water, and performs its task admirably. It is usually added to a batch of nitro to the extent of one part nitric acid, two parts sulfuric acid. Fuming sulfuric acid works even better, but is expensive. This topic will be covered in more detail in the sulfuric acid section.

Other chemicals have been used to soak up water in these batches, but they are not so favoured as concentrated sulfuric acid, because they cost too much. An example of such a substance is phosphorus pentoxide (P2O5) which reacts with water to make phosphoric acid (H_3PO_4).

The last easy method to control the amount of water in the reaction brew is to use as concentrated a sample of nitric acid as possible. This is because in nitric acid, whatever isn't pure nitric acid is water. For example, the usual concentrated nitric acid is 70% nitric acid and 30% water. Nitric acid called fuming nitric acid is 90% nitric acid and 10% water. The drawback to using the more concentrated forms of nitric acid is their cost, and their ease of availability. These last two factors are often enough to rule out their use. More on nitric acid in the nitric acid section.

The temperature at which the reaction is done is the next vitally important area of concern for the explosives manufacturer. There is great danger of not only losing the batch if the temperature of the batch is allowed to climb too high, but also of a disastrous explosion being the result.

At first glance, it may seem simple to keep the temperature of the reacting mix within the proper range. This impression can be of great danger to the novice experimenter. This is because the reaction producing the product makes a good deal of heat as it is progressing. If this heat does not have a chance to escape, regrettable consequences will ensue. There are some very simple tactics to follow to conquer this difficulty.

The first step in keeping reaction temperatures under control is to keep the acids used in the reaction in a freezer before use. This will cool them down well below the temperature required for any explosives production, and will make for quicker production because it will not be necessary to sit around and wait for the acids to cool down in an ice bath, because they will already be cold when they go into the ice bath. Some heat will be made by mixing the acids prior to use, especially if fuming sulfuric acid is used, but the experimenter will still be miles ahead by keeping his acids in a freezer. He will get the added benefit of helping his nitric acid to keep better than it would sitting around. Cooling glycerin and ethylene glycol down in a freezer before use is also good if nitro or ethylene glycol dinitrate is to be made.

Another important technique used to keep the temperature of the reaction cool enough is to add the alcohol to the acids slowly in order for the heat produced in the reaction to be given off gradually. For example, in the production of nitro, the glycerin is added slowly to the acid, taking a break between each addition of the glycerin. In this way the heat produced can be given off to the ice bath surrounding the flask with no problem. Exactly how fast the alcohol can be added depends on how large the batch is. I recommend mentally dividing the alcohol up into ten parts and adding one tenth at a time.

Good stirring is a crucial part of the temperature control strategy. This is because if there is not stirring while the ingredients are being added; local areas where the temperature is too hot will develop in the mix. With stirring, the heat made in the center of the mix will be able to find its way to the walls of the flask and from there to the ice surrounding it. Good stirring has the added benefit of bringing all the ingredients into contact so they can react. This is crucial to getting good results.

Choosing a good vessel to make the explosive in is another aspect to the temperature control problem. The walls of the vessel should not be so thick that they act as an insulator to the flow of heat from the batch into the surrounding ice bath. For this reason, many heavy glass measuring cups are not suitable for use as reaction vessels. This insulation problem becomes more intense the larger the batch of explosive that is being cooked, because of the greater amount of heat given off.

On the other hand, measuring cups have a lip on their rims for pouring that comes in very handy in the production of explosives because the usual procedure for making nitric esters calls for the batch to be poured into water or through a filter when the reaction time is up. This little spout on the rim makes this part of the operation much safer and less messy than using a mason jar, for example.

An ideal reaction vessel, therefore, would be made of reasonably thin glass (about as thick as the usual drinking glass) and have a lip on the rim for good pouring. When making a batch, it should be no more than half full. This is so that good stirring can be done on the contents, and so that the top of the batch can be below the level of the surrounding ice bath. Glasses of this type can be found at fancy bars or in some stores.

Choosing a good thermometer to monitor the temperature of the batches is an area which cannot be ignored. A laboratory type thermometer, of course, will work just fne, but many kitchen thermometers will not. Any portion of the thermometer which comes into contact with the batch must be completely covered with glass. Metal probes will not do because of the strong acids used in the production of the nitric esters.

In a pinch, the typical, cheap, outdoors type thermometer can be used. Here's how: This cheap type of thermometer is just a glass tube filled with mercury attached to a metal backing that has the temperatures printed on it. Generally, one side has øF and the other side has øC. One just takes such a thermometer, and with a three sided file, nicks the glass thermometer body at the maximum-temperature allowed for the reaction it is going to be used in. The glass thermometer body is then removed from its metal backing. It can be used as a stirrer, so long as care is taken not to bang it around the inside of the glass reaction vessel when a batch is being made. As long as the mercury stays below the level of the nick, one knows that the maximum temperature is not being exceeded.

One last major area of potential trouble for the production of the nitric esters must be warned about this is nitric acid that has gone bad, and developed a reddish tint. This is bad news because it means that the nitric acid has broken down into nitric oxide (NO_2) fumes. This acid will not work well for the production of explosives, and may very well cause a disaster. In the production of nitro, for example, it makes the reaction very difficult to control. Bubbling geysers of nitric fumes and detonation can result. How to prevent this and how to treat the problem in progress will be covered in the nitric acid section and the nitro chapter.

Once the ingredients have been mixed and the required amount of reaction time has passed, the most difficult and important part of explosive production must be tackled. This is the purification of the reaction mixture into a pure, refined product. The casual observer may be surprised that the purification of these materials is of such importance. However, it is an accurate assessment to say that the mixing and reacting of the chemicals to make the nitric ester explosives is pretty easy. It is in the purification of the product that the batch is made or broken.

There are two routes to disaster for improperly purified explosives. Route number one, often taken by improperly purified nitro and its close relatives is the least dangerous. With nitro, at least in small quantities, if all the acid used in its manufacture is not removed from the product, then it begins to break down. Within a day or two, it is no longer explosive. In all probability, it reverts back to glycerin.

Route number two, taken by nitromannite and nitrocellulose, to name a couple, is much more dangerous. In these explosives, if all the acid is not removed from the product, the explosive becomes very sensitive and liable to explode unprovoked. At higher temperatures, they become wildly unpredictable. For these reasons, great care must be taken in the purification stage of explosive processing. The directions given in this book will result in pure, stable substances so long as reasonable care is taken. The point is to make the reader aware of the great danger involved in cutting corners on the purification of these materials.

With the major aspects of explosive manufacture discussed in a fairly general way, it is now time to move on to a specific example to illustrate the points that have been made. The example that will be used is methyl nitrate. This choice is not to be construed as an endorsement for methyl nitrate. On the contrary, methyl nitrate is an inferior explosive. While it is very powerful and made from very simple materials, it suffers from twin liabilities that make it unsuitable for most uses. Strike number one is that it is difficult to purify. It must be distilled to reach a good state of purity. This is definitely not recommended. In fact, it is a good recipe for a devastating explosion.

Strike number two is that the stuff evaporates away too easily. This will ruin any gelatin or plastique that it is incorporated into because even in the bound up form of gelatin or plastique, the methyl nitrate will evaporate away, leaving nothing but the binders and fillers. The only way to stop this is to keep it in a freezer until use (thereby cutting evaporation to almost nothing because it is too cold to evaporate) or to seal the explosive in a container that will hold in the vapours against the pressure they will generate at normal temperature. A good choice for such a container is a champagne bottle with the cheap plastic stopper.

Strike number three, as if any more were needed, is that this explosive is very sensitive. In many respects, it is more sensitive than nitro, without the many redeeming graces of nitro. The one good point about this material is that the crude stuff before it is distilled (danger!) is very sensitive to heat, and can be used as a heat detonated explosive.

PREPARATION OF METHYL NITRATE

This is about the most complicated procedure used in making any of the nitric esters. If you feel confused when reading this, do not be discouraged. Many of the better explosives are made much more simply. The only special piece of chemical glassware that would come in handy for this preparation is a separatory funnel (unless distillation of the crude product is attempted explosion DANGER!). A good eyedropper can replace the separatory funnel in this procedure. I'll explain how in the text.

Methyl nitrate is made by reacting methyl alcohol (methanol, available very cheaply at the local hardware store) with concentrated nitric acid (70% nitric acid, density or specific gravity 1.42). The water absorbing chemical in this reaction is concentrated sulfuric acid.

$$CH_3OH + HNO_3 \xrightarrow{H_2SO_4} CH_3ONO_2 + H_2O$$

To begin preparation of methyl nitrate, all three ingredients, methyl alcohol, sulfuric acid, and nitric acid are cooled down in a freezer. Then a plastic or styrofoam tub is filled half full of ice. This is the ice bath which will be used to keep the reaction cold while it is being done. Next a 5 gallon plastic pail is filled 2/3 full of cold water, and placed next to the tub of ice. This pail is the preliminary disposal site for the used acids after the reaction is done, and also serves as the emergency safety dump to toss in runaway reactions before any serious damage is done. Finally, the ice is wetted with some water to make it work better at cooling down the reaction vessels.

Now the cold ingredients are measured out. With a glass measuring cup, measure out 300c.c. of nitric acid, and 300c.c. of concentrated sulfuric acid. (Note: I c.c. = 1 ml. It is also important that the nitric acid not have a reddish tint to it.) The nitric acid and sulfuric acid are then mixed together in a measuring cup. The cup filled with the mixed acids is then placed in the ice bath to keep it cold. In a second measuring cup nestled well within the ice bath, put 150 ml of methyl alcohol. Then to the alcohol, add 50 ml of concentrated sulfuric acid. This must be done slowly, with good stirring so that the temperature of the mix does not go above 10ø C (50ø F).

Next three glass reaction vessels are put into the ice bath. They should be about one pint in size (500 ml) have a pouring spout on them and preferably be tall and thin like a drinking glass. Into each of these glass reaction vessels is put one third of the nitric acid-sulfuric acid mix.

With all in readiness, the methyl nitrate can be made. The most conveniently located reaction vessel is picked out, and to it is added 1/3 of the methyl alcohol-sulfuric acid mix. One third amounts to about 60 ml because of contraction of the solution during mixing. This addition must be done slowly with good stirring. Care should be taken not to bang the glass stirring rod against the side of the reaction vessel during stirring. The temperature of the mix is allowed to go up to 40ø C (104ø F) fairly quickly, and then kept at this temperature by cooling in the ice bath. The rate at which the temperature rises can be controlled by how fast the ingredients are added.

When the full 60 ml has been added, the stirring is continued for another minute or two, then stopped. The product, methyl nitrate, rises to the top of the liquid as a clear oil. If one has a separatory funnel, one can wait for another ten minutes or so for the reaction to finish, then pour the reaction mixture into the separatory funnel, let it sit for a minute or two for the methyl nitrate layer to float up again, then drain off the acids into the 5 gallon pail and pour the methyl nitrate product into a small glass container that already has 20 ml of cold water and 5 grams of table salt in it. This salt water is the first step in the purification of the product.

I must warn here against touching methyl nitrate or tasting it, because it will cause terrible headaches. Too much smelling of the product will cause the same effect.

If one does not have a separatory funnel, one can improvise. Once the mixing of the ingredients is done, and the product comes to the top, it can be sucked up in an eyedropper and squirted into the salt water. This must be finished before the ten to 15 minute time limit is up, because after that time great geysers of nitric oxide fumes are gushed up by the acids. They must be dumped into the pail of water by that time. Once the water dilutes them, the danger is past.

Nitric oxide is a pretty good poison. Breathing its fumes can easily lead to a delayed death. It burns the insides of the lungs, which then fill up with fluids leading to death. Symptoms often do not come on for a day or two. Whenever a batch starts to bubble up red fumes, it must be drowned in the pail of water to avoid severe danger!

With the first reaction vessel finished, one can then move on to the second one, and then the third one. The product layers from the second and third vessels are put in with the salt water wash that the product from the first vessel is sitting in. This salt water wash should be swirled around from time to time as the production is continuing to aid in getting traces of acid dissolved in the water. Since traces of acid are the enemy here, it is important that only the methyl nitrate layer be put into the salt water, not the acid it is floating on. One must take some care in separating the layers, or in sucking up the product layer with an eyedropper.

The salt water wash bottle has about 175 ml (a little over 200 grams) of methyl nitrate in it. It should be swirled around some more, then allowed to sit for a few minutes. If one has a sep. funnel, it should be poured into it, and then the water layer (the bottom one) is drained off. If an eyedropper is being used, the water layer should be removed with it.

Now the remaining acid in the crude product must be removed. To do this, some lye (a volume amounting to about 5 match heads) is dissolved in about 50 ml of water. This lye water is then mixed in with the crude methyl nitrate and allowed to react for about 10 minutes with some more swirling. At the end of this treatment, a drop from the water layer should still be basic (it should turn red litmus paper blue). Litmus can be obtained at the local drug store, or at any lab or chemical supply outlet, with no suspicion.

I cannot overemphasize the importance of removing the acid from the crude product. Traces of acid left in it will cause it to break down and become so unstable that it becomes too dangerous to be of any use. If one has a sep funnel, gentle shaking can be done for a few minutes to make sure that the methyl nitrate gets into good contact with the lye water. If one is not at hand, the stirring with the lye water must not be a slipshod job. Again I must emphasize that at the end of the lye water treatment, the lye water should still be somewhat basic. If the water is acidic (turns blue litmus red) a few more match heads worth of lye must be added, and the treatment continued.

When the lye water treatment is completed, the final purification of the product is next. The lye water is drained off by use of a sep funnel or eyedropper, and then 30 ml of ice cold salt water (5 grams of salt) is mixed in with the methyl nitrate. After a few minutes of gentle shaking in the sep. funnel, or good stirring, the mix is allowed to sit. The salt water will contain most of the unused lye that happened to find its way into the methyl nitrate. The salt water layer is separated, and thrown away.

Finally 30 ml of ice cold water is added to the methyl nitrate, and once again, stirring or shaking is done. The methyl nitrate is now practically pure, except for some water dissolved in it. The water layer is separated off of the methyl nitrate, and the product is ready for immediate use. The small amount of water dissolved in it will cause no real problems so long as the product is used soon after making it. A more stable product results if it is distilled, but the great danger in doing this can't be underestimated, especially for the unskilled. The product distills at 65ø C. Heating must be gradual, and an oil bath must be used to heat the flask. The temperature of the oil bath must not get much above 65ø C, or the contents of the distilling flask will superheat, leading to an explosion.

With the product at hand, storage can be done by pouring it into a tightly stoppered bottle and placing it in a freezer. This material should not be stored for more than a few days before use. Methyl nitrate is a use-it-as-you-make-it explosive. The great friction sensitivity of this substance means screw type bottles can't be used. All stoppers should be well greased with vaseline to reduce the danger from this source of friction.

Methyl nitrate becomes increasingly sensitive as its temperature is raised. It also does not age well. These facts are especially important for a product which has not been purified by distillation. One can take advantage of these facts by using methyl nitrate as a heat and vibration detonated explosive. For example, a half pint bottle (like a used liquor bottle) loosely attached with heat resistant tape to the exhaust manifold of an automobile is very likely to destroy the offending vehicle after a short warm up period. Similarly, a bottle which finds its way into an incinerator is very likely to destroy the incinerator.

More certain detonation can be obtained by leading a section of nichrome wire (toaster heating element) into the liquid. When current flows through the wire, the red hot heat of it will cause detonation. The variety of diabolical booby traps which can be constructed around this principle is limited only by the imagination.

A more convenient and safer form of methyl nitrate is gelatin. Gelatin is very easy to make from methyl nitrate, and is almost as powerful as the pure explosive. To make gelatin, a little bit of guncotton (aka nitrocellulose or smokeless powder) is mixed in with the methyl nitrate. A stiff jelly quickly forms which can be used as a plastic explosive. The resulting jelly is even more powerful than C-4. The keeping properties of this gelatin, however, are no better than the pure methyl nitrate. It must be used soon after production.

Since the production of gelatins by mixing a liquid explosive with guncotton will be seen repeatedly in this book, something must be said about guncotton. There are a wide variety of sources one can go to, to get guncotton. For example, cans of guncotton can be obtained at sporting goods stores. This material is stocked because some people "reload" their own ammunition for higher performance or to save money, or just to have fun. Large drums of guncotton (labelled nitrocellulose, with a second rating which tells how finely divided it is) can be found at any factory which makes varnishes. Nitrocellulose is a key ingredient of varnish. This material works great, but it comes packaged in the drum soaking wet with isopropyl (rubbing) alcohol because it is safer in this state. The alcohol must be allowed to evaporate off before it can be used to make gelatin. Guncotton can also be obtained by taking apart bullets or shotgun shells. In this source, however, as with the "reloading" supply route, one must avoid buying what is called "double based" guncotton. Double based guncotton has a few percent nitroglycerin added to it to give it an added punch. This material should be avoided unless one is prepared to do the necessary calculations to compensate for the nitro already in the product. 12 gauge shotgun shells almost always have a double based propellant.

To make the gelatin, one first measures out how much methyl nitrate is going to be jelled. This can be done by weighing it on a scale, or measuring its volume in ml and multiplying by 1.2. The result will be its weight in grams. The methyl nitrate is then poured into a plastic dish. Then a quantity of guncotton amounting to one tenth the weight of methyl nitrate is weighed out. For example, with 250 grams of methyl nitrate, one weighs out 25 grams of guncotton. The guncotton is then added slowly to the methyl nitrate with gentle stirring with a glass or wooden rod until a uniform mixture results. Alternative mixing bowl-mixer combinations would be; stainless steel bowl with plastic or rubber stirrer, or glass bowl with wooden stirrer. Avoid the plastic bowl-rubber stirrer combination as this could generate static electricity, and a tiny spark could set the guncotton off.

The resulting fairly stiff jelly has 9% guncotton, and will be set off with a number 4 blasting cap when it is fresh. The use of a little bit less guncotton produces a much more sensitive jell which can be set off with the weakest of all caps, a number 1. A good firecracker will set off the pure methyl nitrate.

This gelatin has a severe drawback that the other gelatins do not have. The methyl nitrate will evaporate out of it on storage. This can be slowed up by keeping it in a freezer until use. Storage must be very short-term because of the bad keeping qualities of the methyl nitrate. It should be noted that frozen gelatin is more sensitive than gelatin at normal temperatures. Avoid screw type lids. I recommend a zip lock baggie.

Before moving on to the good explosives, some discussion is called for here on an alternative process for making methyl nitrate, and on the general topic of distilling methyl nitrate. As you saw repeatedly pointed out in the above text, one of the big drawbacks of methyl nitrate is that it does not store well unless it has been distilled. An alternative process for making methyl nitrate centers around distilling the product directly out of the reaction mixture (i.e. the methyl alcohol-nitric acid-sulfuric acid mix), thereby avoiding a lot of the washing hassles that are the main sticking point of the production method given here.

This method has obvious advantages for industrial-scale production because it is faster and simpler. For this reason, it was adopted by the Nazis in the closing days of WW II to make methyl nitrate. Before rushing off and following their lead, keep in mind that life was cheap in Nazi Germany, especially the lives of industrial slave labourers. Even in skilled hands, the distillation of this material is not to be taken lightly.

The foremost precaution which must be taken in distilling methyl nitrate is to have all traces of nitrous acid removed from the mixture. This is done by adding urea to it. The fertilizer grade of urea will do for this purpose, but before rushing off and tossing any nitrogen fertilizer into the brew, make sure that the nitrogen fertilizer is made of urea, and not the much more common ammonium nitrate.

The second precaution is to prevent overheating of the mixture while distilling it. There are a couple of angles to approach this problem from. Step number one is to heat the distilling flask using an oil or hot water bath. Direct exposure to the source of heat is a no-no because that will cause local overheating and result in an explosion. The temperature of the bath should be just a few degrees higher than the boiling point of the product. At normal atmospheric pressure, the boiling point is 65ø C.

A more effective method for keeping the temperatures safe during a distillation is to distill under a vacuum. There is a problem with this approach because the normal boiling point of methyl nitrate is not very high to begin with. If it is lowered very much by applying a strong vacuum, a condenser, or even a collecting flask packed in ice will not suffice to turn it back into a liquid. With the strong vacuum that an aspirator or laboratory vacuum pump produces, dry ice would be required to condense the product. For those not intimately familiar with vacuum distillation, I must explain that the boiling point of a substance rapidly falls off as the vacuum becomes stronger.

The vacuum distillation problem can be solved if the source of vacuum is not very strong. One wants to reduce the pressure inside the flask to about half normal pressure (a vacuum of 15 inches of Mercury). This will lower the boiling point of the substance 15 or 20ø C, making it about as hard to condense as ether.

A good source of such a weak vacuum is a water bed drainage pump. This T-shaped plastic vacuum source produces just the right vacuum for the job when cold water is flowing through it. Alternatively, cheap workshop pumps will do, although one must be wary of the fumes of methyl nitrate they will exhaust into the air. The water bed pump is far superior for the job.

The condenser must also be efficient. My recommendation is a Graham condenser which has ice water siphoned through it as a coolant.

The Graham condenser has a spiralled central tube for maximum efficiency. Again, I must warn that this method is dangerous and should only be attempted by skilled professionals.

NITRIC AND SULFURIC ACIDS

Before moving on into the manufacture of some of the really good explosives, it would be best to make the reader familiar with the two ingredients which will be used over and over in the processes described here. They are nitric and sulfuric acid. A familiarity with these two substances will take much of the mystery out of explosive manufacture.

NITRIC ACID

Nitric acid is the key, indispensable ingredient in the manufacture of the explosives found in this book. The sulfuric acid can often be made do without, if one is willing to use a greater amount of the most concentrated nitric acid, and accept a lower yield of product in the process. Not so with the nitric acid. Only nitric acid in the specified or higher strengths will give that desired product.

With the premium thereby placed on nitric acid, it is a fortuitous circumstance that nitric acid is such an important industrial material with a wide variety of uses ranging from the exotic to the mundane. It can be found in the pint to quart range in any chemical laboratory (most abundantly in those labs doing metal analysis) and in the 55 gallon drum volume in plants involved in the manufacture of explosives(!), fertilizers, dyes and fabrics. Jewellers and others involved in trading or handling precious metals often have some on hand, too, because the best test for determining whether something is made of the "noble metals" (i.e., gold or platinum) is to apply some nitric acid to the metal and see if there is any reaction. Only the noble metals stand up to nitric acid. A further test mixture such as a jeweller is likely to need is aqua regia. This is a mixture of nitric and hydrochloric acid (mixed on the spot, not purchased mixed) and is the only liquid which dissolves gold or platinum.

Nitric acid can be obtained in reasonably large amounts, by anyone who does not look like a refugee from the local insane asylum or drunk tank, at drug stores or chemical supply outlets. In most cases, a drug store will have to send out for it, and small chemical suppliers may have to also. Fuming nitric acid is less likely to be on the shelves than the standard concentrated nitric acid.

Mail order outfits are another useful source for nitric acid, and for that matter, sulfuric acid. These two acids, along with hydrochloric acid, are so widely used that no serious suspicion can be put upon the buyers of just these materials. There was a scandal a few years back concerning mail order chemical outfits being set up by the DEA to entrap wanna-be drug chemists, but to the best of my knowledge, their games did not extend to the explosives field. A possible exception to this is a chemical supplier I saw advertise in Soldier of Fortune magazine a few years ago offering "explosives manufacture chemicals." Such brazen advertising is cause for suspicion. My judgement on the matter is that since neither nitric nor sulfuric acid play a central role in "controlled substance" manufacture, orders for these materials should raise no eyebrows in these dope-crazed times.

Nitric acid is a liquid that should be clear if it is the 70% grade, and yellow colored if it is the 90% grade. The color in the higher strength acid is caused by the acid breaking down to NO_2. This small amount of breakdown can be tolerated in most instances. The condition that cannot be tolerated in either strength of acid is when the acid takes on a pink or reddish brown color, and a cloud of reddish gas can be seen inside the bottle of acid. In this case, it has broken down so much that it can't be used for manufacturing explosives. This breakdown process can be largely stopped by keeping the acid cold and in the dark. A freezer meets these conditions nicely. Being careful to keep traces of dirt out of the acid bottle or jug is another big step towards keeping the contents in a usable condition.

Occasionally, directions will call for what is called "white fuming nitric acid." This is the 90% fuming nitric acid which has been treated with urea to remove the traces of NO_2 and the yellow color that comes with small amounts of it. Urea can also be used to clean up nitric acid that has a darker color to it, but a point comes where this should be considered futile. When the red cloud shows up above the surface of the acid, it should be considered ready for flushing down the toilet. The procedure used to knock out the NO_2 contaminants calls for adding a little bit of urea to the nitric and, stirring it in and warming the mixture gently. If the color persists, dry air is blown through the liquid, and if there is still color, some more urea is added.

As was hinted at before, the 90% (or higher) strength fuming nitric acid is not so easy to casually pick up as the standard 70% concentrated nitric acid. It is also much more expensive to get the fuming acid instead of the 70% acid ($7 per pint vs. $35 per pint). For these reasons, it is sometimes advantageous or necessary to make the high strength acid oneself.

The procedure for making high strength nitric acid is pretty simple, but some chemical glassware is called for to do a good job. Various pamphleteers have included directions for making high strength nitric acid in their books, leaving out a crucial aspect of the process. This crucial aspect is that the nitric acid formed must be distilled under a vacuum. Without the vacuum, a reddish colored product is made that is not very suitable for use in making explosives.

To make this good quality high strength acid, the following materials are needed: concentrated sulfuric acid, sodium nitrate, and dry ice. Also required is a distilling kit with ground glass joints and a vacuum adapter so the product can be distilled under a vacuum. The best vacuum source is an aspirator (cost: $10) because the acid fumes will not harm it, and the water flow will flush them down the drain.

The distilling kit is a substantial investment at a few hundred dollars, so one should not be purchased unless it is going to get enough work to make the investment worthwhile. Laboratory supply companies will either carry the equipment or can get it in a few days. Check the Yellow Pages. The ground glass joints must be greased up with stop-

cock grease before assembling the glassware to keep the joints from getting frozen together, and to make for a good vacuum seal, free from leaks. No parts may be made of cork or rubber, because the nitric acid will destroy them, and become polluted in the process. Only glass or Teflon parts are allowed.

Now to begin. Into the 2000 ml distilling flask is put 685 grams (365 ml) of concentrated sulfuric acid. Then 600 grams of sodium nitrate is mixed in with the sulfuric acid. Simply swirling the flask as the sodium nitrate is being added will do an acceptable job of mixing. Sodium nitrate is pretty easily available. I always used to find it on the shelves of my local Walgreens store, labelled saltpeter (technically this is the wrong name because saltpeter is potassium nitrate). It sat on the shelf right next to sublimed sulfur. Very interesting combination.

There will be no obvious reaction as the two ingredients are mixed. The glassware is assembled as shown in the diagram, and the 1000 ml receiving flask is packed in dry ice (styrofoam tub suggested) and the dry ice is wetted with rubbing alcohol. Now water is turned on to the aspirator. (See a college level organic chemistry lab manual for more on aspirators, but they work just like water bed drain pumps, only better. The threaded end is the water inlet, the water comes out the opposite end, and the vacuum is produced at the side arm of this T-shaped device.) The water must be cold for good results. An automotive type vacuum hose is led from the side arm to the vacuum nipple on the vacuum adapter, and within seconds a strong vacuum develops inside the glassware. It should be strong enough that the glassware can't easily be disassembled. Eye protection is required as always when dealing with caustic chemicals.

Some bubbling will begin in the 2000 ml flask as nitric acid begins to boil out of it. Heat should be applied from the hot plate to keep the process going. A good, but not violent rate of boiling is what the doctor ordered. In about 20 minutes, close to 300 ml of pure nitric acid will collect in the dry ice cooled flask. It will freeze shortly after its arrival there. Since nitric acid contracts on freezing, this poses no danger of breaking the glass. The heat applied to the distilling flask should be gentle. There is no need to make everything red hot. When the bubbling of the distilling flask slows, and the expected amount of product appears in the receiving flask, the heat is turned off, and the vacuum hose removed.

As soon as the distilling flask cools off, it is advisable to rinse out the distilling flask (it contains sodium hydrogen sulfate, a solid often used as a drain opener) and fill it with a fresh load of sodium nitrate and sulfuric acid. By repeating the vacuum and heating on this load, another 300 ml of product will come over to the 1000 ml receiving flask. Repeating the process yet one more time will leave the receiving flask nearly full. This is a good time to break. A quart of fuming nitric acid is enough to produce a very useful amount of explosive. Store the product in the freezer until use.

Before leaving this topic, I should mention one more thing about the cheap drug store grade of sodium nitrate ("saltpeter"). The product I always found at Walgreens was nearly soaking wet. In this state; it was useless for making gunpowder and not so good for making nitric acid either. Before using a wet material in this process, spread it out on a Teflon coated cookie sheet or a glass pan and bake it in the sun on a hot dry day. Do not bake it in the oven because too much raw heat will break it down into the red poisonous gas, NO_2. A microwave oven may be OK for drying this material. I suggest this for serious experimenters.

One more tip is called for on the nitric acid production process. The best type of vacuum adapter for use in the distilling set-up shown is one that has a drip tip that extends past the ground glass joint. This will ensure that the nitric acid fumes make it down into the flask and are condensed and frozen there. Too short a drip tip can lead to the acid getting condensed in the area of the ground glass joint, and if it freezes there it could plug up that part of the apparatus. This will choke off the source of vacuum to the glassware, and may even block the flow of nitric acid into the receiving flask.

SULFURIC ACID

Like nitric acid, sulfuric acid is an extremely common chemical, both in laboratories and in industry. Its wide variety of uses makes obtaining sulfuric acid a very easy task. For example, any shop doing electroplating (especially plating chrome) will go through large amounts of sulfuric acid. Plating shops also use a lot of nitric acid, especially if they are plating aluminium objects. It is easy then to see how a plating shop can be a one-stop shopping center for all one's acid needs.

Home hobbyist electroplaters need these acids as well, so this can be a good and believable cover story for anyone who runs into the embarrassing situation of being asked what the materials are needed for by a nosey druggist or other small-time chemical supplier. Mail order outfits may also be considered safe for ordering sulfuric acid from because of the commonness of the chemical and because it plays no central role in drug manufacture.

This is by no means a complete list of all the places that use sulfuric acid and are likely to have it on hand. Virtually every large-scale manufacturing process uses sulfuric acid at some point or another. A much more important question is: Will the material at hand be good enough for use in explosives manufacture? Here let the reader recall the great importance of keeping the amount of water present in the reaction mixtures to an absolute minimum, lest the yield of product be reduced to nearly zero. With this in mind, it should then be obvious that battery electrolyte sulfuric acid is not acceptable because it is half water. In theory, the water can be cooked away, but this produces dangerous fumes and results in an inferior product.

Similarly, a lot of industrial sulfuric acid has a strength of around 80%, with the other 20% being water. This grade will not do, either. The materials that are acceptable are concentrated sulfuric acid, or fuming sulfuric acid. Fuming sulfuric acid will be clearly labelled as such and may list what percent by weight SO_3 (aka oleum) it contains. More on this later. Concentrated sulfuric acid may only be recognizable as such by a density (or specific gravity: sp. gr.) reading printed on it. Concentrated sulfuric acid has a density of 1.85 to 1.91, whether this value is given in pounds per pint or grams per ml.

Both the fuming and concentrated acids are likely to be kept in glass bottles, jugs or carbouys because these corrosive acids have bad effects upon metal. Plastic containers are another possibility.

As was mentioned earlier, there are two useful types or grades of sulfuric acid. They are concentrated sulfuric acid (H_2SO_4 and fuming sulfuric acid ($H_2SO_4\,SO_3$). To get an understanding of the difference between the two, one should look to how sulfuric acid is made. Huge plants produce the gas SO_3 (sulfur trioxide) by burning sulfur to SO_2, and then inducing it to pick up another oxygen atom by high heat and catalysts to form SO_3. SO_3 reacts with one molecule of water to form pure sulfuric acid. So these plants just mix the SO_3 they make with water and turn out pure concentrated sulfuric acid.

If they leave in a little extra SO_3, (i.e., they don't add enough water to completely turn the SO_3 to sulfuric acid), fuming sulfuric acid results. This fuming sulfuric acid is even better at soaking up water from one's explosive batches. First it scavenges furiously for enough water to turn its SO_3 to sulfuric acid, and then when this is accomplished, the resulting concentrated sulfuric acid picks up from there. So fuming sulfuric acid is a dehydrated sulfuric acid that is even better at soaking up unwanted water than concentrated sulfuric acid.

Fuming sulfuric acid, like fuming nitric acid, is aptly named. The stuff actually does fume. These fumes are very dangerous to breathe in because they form sulfuric acid with the moisture in the lungs and throat, producing acid burns. For the same reason, rubber gloves must be worn when handling fuming sulfuric (or nitric acid) acid because the fumes will likewise burn any skin they come in contact with.

Whenever a fuming acid is to be used in a reaction, it is very important that good ventilation be provided. The experimenter should be upwind from the chemicals to ensure safety. This is most important when measuring out the chemicals, and during the early stages of the reaction. Once the chemicals have had a chance to react together for a while, and the acid loses some of its strength, the danger from acid fumes decreases.

There is no really good or practical method for the home experimenter to make his own sulfuric or fuming sulfuric acid. It would be way more bother than it is worth, and the product is more than likely going to be of an unacceptably low grade. Sulfuric acid should either be purchased through the previously mentioned commercial sources, or obtained from labs or industries. Good sulfuric acid is clear as water and is almost as thick as lightweight motor oil. Prices vary with the outlet, but one can expect to pay in the neighbourhood of $15 per gallon for concentrated sulfuric acid, and $35 per pint for fuming sulfuric acid.

Sulfuric acid keeps really well, so it needn't be treated so gently as nitric acid. Light and heat have no real effect on it. It is advisable to cool it down in the freezer before use to cut down on the amount of time which would otherwise have to be spent cooling the ingredients before they are mixed to react. The only important storage tip is for the caps to be kept tightly closed on fuming sulfuric acid so the SO_3 fumes do not escape into the air.

NITROGLYCERIN

If one was for some reason forced to choose a single all-purpose explosive or if one were to wish away all the explosive substances save one, nitroglycerin is the chemical to clutch close to one's heart. This popularly, and for the most part unjustly, maligned explosive is so powerful, versatile, and easy to make that it is far and away the number one choice for a Home Workshop Explosive manufacturer.

We've all seen the Hollywood hogwash a million times where the hero thrusts himself into "mortal danger" with nitro. Small droplets of nitro oozing from old sticks of dynamite fall to the ground and explode on landing, like souped up firecrackers. Let me tell you right up front that this is the purest form of bull. Some of my fondest adventures centered around nitro, and it is nowhere near that easy to set off. There is no reason why anyone who takes reasonable care and does not suffer from at terminal case of the shakes cannot handle nitro safely. I have had occasion to fall in a drunken stupor on broken sidewalks, and my vial of nitro land on the sidewalk next to me. No explosion. I have dropped quantities of nitro from a height of several stories and had it land in a couple inches of snow with no detonation. It has been my experience that so long as one works with fairly limited quantities of nitro at a time, and then processes the product into gelatin or plastique, the dangers one faces are minimal and manageable.

The need for small batches may at first seem discouraging for those special applications where a large amount of blasting power is required. It should not be. This drawback can be conquered by running one's batches serially. A large part of the nitro manufacturing process is sitting around waiting while the various cleansing operations work. This dead time can be profitably filled by starting another batch to feed into the clean-up section of the operation. It has been my experience that a couple ounces of product can be routinely run with none of the complications that can arise from runaway reactions with larger loads. Since only about 45 minutes is needed to do a batch, a healthy rate of production can be maintained. With a competent and trustworthy helper at hand, these serial production techniques are vastly simplified. In the production section of this chapter, I'll provide my suggestions on how to organize this serial batch production effort.

Now it is time to delve more deeply into exactly how nitro production is set up and why one should regard nitro as the explosive of choice.

To answer the last question first, one only has to consider the extreme simplicity of making and purifying nitro. It was first made in the middle 1800s. This speaks volumes when you consider the crude materials and equipment available at the time. It has been my experience that anyone who is not brain damaged or terminally stoned can easily master the process. So long as attention is paid to following the directions, there is virtually nothing that can go wrong.

Nitro is a member of the nitric ester family, and is made the same way as the rest of the family. Nitric acid is reacted with an alcohol (in this case glycerin) to form the ester nitroglycerin.

Sulfuric acid is added to the mixture to soak up water, and there by increase the amount of nitro produced.

$$3\ \text{Nitric Acid} + \text{Glycerin} \xrightarrow[\text{Acid}]{\text{Sulfuric}} \text{Nitro} + 3\ \text{Water}$$

Both sulfuric and nitric acid have already been covered, so no more will be said about them other than to say that both concentrated and fuming nitric acid can be used to make nitro. Same with the sulfuric acid, both concentrated and the fuming acids will work for making nitro. The procedures are a little different depending on what strength acids are being used, but that will be covered in detail in the production section of this chapter. Let me just say up front that the best yields of product are to be had when one of the acids used is of the fuming grade. It doesn't matter which one of the acids is of fuming strength, so cost or ease of availability is the overriding factor in choosing which acid to get in the fuming strength. Results are better with the fuming grade because of the smaller amount of water added to the reaction mixture.

Glycerin is the other ingredient for nitro manufacture. As luck would have it, it is very easy to find. Look on the shelves of any drug store, and you will find it. They have it in little bottles for use as an "emollient" to help dry skin. The problem with just going to the drug store and picking out a few bottles of whatever is handy is that many brands of glycerin are cut with water. Since glycerin is a clear liquid, it doesn't show up just by looking at it.

The first step to bypassing this potential pitfall is to pick up a few different bottles of the competing brands, and check them for their water content. The easiest way to do this is to see how runny the glycerin is. Adding water to glycerin makes it runnier. The difference can be made more obvious by cooling the bottles down in a refrigerator. Good glycerin will get thick like honey.

If no brand is clearly superior to the others, one must check to see if all the brands are garbage, or if they are all good. To do this, the glycerin is cooked to see how much water will boil out of it. The best way to go about this is to pour the contents of a bottle of glycerin to be tested into a glass measuring cup (best if it is made out of pyrex or kimax so it will withstand the heating) and then place the glass measuring cup into a small pan or other metal container that is filled with enough cooking oil to reach about half way up the sides of the measuring cup. This is placed on a stove and heated. The temperature of the glycerin is monitored by use of any convenient sized cooking thermometer. The glycerin should be stirred regularly to make sure that it gets heated evenly. There is no danger in this process since glycerin is no more unstable than the cooking oil. When the temperature of the glycerin reaches

100øC (212øF), boiling may be noted in the glycerin. If it does boil, that means there is water in it. Glycerin does not boil until a temperature of 290øC is reached. The heating should be continued until a temperature of around 200øC is reached, and then the heat can be turned off and the contents allowed to cool.

To interpret the results, simply read on the measuring cup how much of the glycerin has boiled away. Some small amount of boiling can be expected because glycerin will soak up water from the air just by being exposed to humid air. If, however, more than a few percent of the glycerin boils away, there is too much water in the glycerin for best results. In making these volume measurements, it is important that the glycerin be at about the same temperature for both the before and after reading. This is because glycerin expands quite a bit when it is heated, so taking one reading while the glycerin is hot will give inaccurate results.

If the glycerin has an unacceptable amount of water in it, one has a couple of choices. The first and easiest choice is to just keep shopping until a good grade of glycerin is found. If you do not strike pay dirt by this method, the more difficult route of distilling the glycerin must be followed.

Industrial manufacturers of nitro almost always distill their glycerin before using it. This puts their worried little heads to rest on the water issue. It is pretty safe and easy to do, but the distilling equipment described in the nitric acid section is needed to do a good job. An alternative is to follow the procedure described just above for testing the amount of water in the glycerin. If the temperature of the glycerin is held in the 150øC range for an hour or so, almost all the water will boil out of it. It will pick up a yellowish color during the heating process due to the formation of acrolein compounds, but this does not cause any harm. Industrial distilling techniques give the same yellow color. It just means that the nitro will be yellow colored instead of the nice clear product that could be had with better glycerin. If this clear product is desired, a vacuum distillation of the glycerin will give it. If the experimenter has experience doing vacuum distillations, this is the best route to follow. I must warn that glycerin has a tendency to bump during vacuum distillations. Adding a healthy amount of glass wool (angel's hair) to the distilling flask will keep this under control. Boiling chips do not do a good job with glycerin.

So with the preliminaries taken care of, it is time to move on to the actual nitro production process. To start with, a 5 gallon plastic pail is filled about 2/3 full of cold water and placed next to the production area. It serves the same purpose that it did in the methyl nitrate section. If in the unlikely event that the batch gets put of control and begins gushing out the red fumes of poisonous NO_2, the batch can be drowned in the water and the danger averted.

Then on a table next to the water pail is placed a styrofoam tub or other suitable container about half filled with crushed ice. Into this ice, a glass vessel is nestled. It should be about a pint in volume and have the thin glass and pouring lip mentioned earlier when describing the ideal reaction vessel.

Into this glass vessel, put 100 ml of concentrated nitric acid and 200 ml of concentrated sulfuric acid. These acids should be cold to start with by being stored in a freezer. They are mixed together by swirling the container, or mixing with a glass rod or thermometer. This mixing will warm them up a little bit, but they will quickly cool again in the ice bath. When the temperature of the acids is under 40øF (4øC), the glycerin can be added. To do this, about 50 ml of glycerin is measured out. It is then added to the acid mixture 5 ml at a time. The best way to do this is to let the glycerin portions run down the side of the glass. This ensures that the glycerin enters the acid mixture gradually rather than in lump sums.

The mixture must be stirred during the addition to keep the glycerin from building up in one particular spot. If it should build up in one spot, a runaway reaction would result. There are several choices for stirring techniques. My favourite is to tilt the container at about a 45 degree angle and then rotate the vessel in a manner similar to a cement mixer. This very gentle technique works well. Another alternative is to swirl the vessel. It could also be stirred by using the glass thermometer. Doing this, it is important not to bang it around or to create friction by scraping it on the walls of the vessel. This is an inferior technique. Nitro factories stir their batches by blowing a stream of dry air through the mixture. I would forget about this one too, and just stick to the cement mixer technique or swirling.

The addition of the glycerin to the acids should take about 25 minutes, adding ten portions of glycerin of about 5 ml each. The temperature of the reaction mixture should be watched during the addition of glycerin. It should not be allowed to rise above 50øF. If it gets too close to this temperature, the additions of glycerin should cease, and the mixing continued with the vessel nestled in the ice bath until the temperature drops back down to around 40øF.

Under no conditions should it be contemplated to just add all the glycerin, nitric and sulfuric acid together all at once "to save time." To add them all together at once will result in a geyser of red fumes in a small batch, and possibly an explosion in a large batch. Likewise, it should not be contemplated to add the acids to the glycerin. This chapter will cover all the good variations on nitro production.

When all the glycerin has been added, a milky colored solution will have been formed with little globules of pure nitro dispersed throughout the mixture. After the glycerin has been added, the mix can be allowed to warm up a little bit. It can be taken out of the ice bath, and its temperature allowed to rise into the 50's F. Stirring should be done occasionally during this period.

About 20 to 25 minutes after the last addition of glycerin, the reaction can be considered to be finished, and purification and processing commenced. The first step in processing the nitro is to pour the whole batch into a little over a quart of cold water. Most of the acid dissolves in the water, and crude nitro settles out at the bottom of the container as an oil. It goes to the bottom because it is heavier than water. A small amount of nitro stays floating on

the surface of the water due to surface tension, but the amount is not that great. Some of the droplets can be sunk by splashing around on the water surface with some type of stirring rod.

The fact that the nitro sinks to the bottom presents the first obstacle in the purification of the nitro. It is obviously going to complicate things getting the nitro off the bottom of the container for further purification. The best solution is to get a 2000 ml separatory funnel. A quart or so of cold water can be put in it, and the batches poured into it when they are done. When the nitro settles to the bottom of the sep funnel, the stop cock can be opened, and the nitro drained out. The leftover acid water can then be poured down the drain. It is important here that the separation between nitro and acid water be as sharp as possible because the whole idea of the purification process is to remove this acid from the nitro.

Another possible method of getting the nitro off the bottom of the container is to tilt it to one side to pool the nitro, and then suck it up with a plastic turkey baster.

Whichever method is used, the separated nitro is then added to about a cup of clean, cold water and allowed to sit there for a while. This clean water will soak up more acid from the nitro. It is good to cause the nitro to flow around on the bottom of its new container every once in a while, so that it is not the same old surface exposed to the water all the time. This water bath should take no more than about an hour.

Next, the nitro must be carefully separated from the water once again, and added to about a cup of bicarbonate of soda solution. The bicarb solution is made by adding about 10 grams of Arm & Hammer to a cup of water, and mixing until it is dissolved. The importance of this step cannot be underestimated. The nitro, even after the water washing it has received up to this point, still contains some acid. If the acid is not knocked out, the nitro will not keep. The breakdown of the nitro is fairly rapid. Within a day of being made, almost all of it will have decayed without this bicarb treatment to neutralize the excess acid in it. With small batches, this breakdown is a quiet process, resulting in a non- or poor explosive mixture. What happens to larger batches is problematic.

When the nitro is added to the bicarb solution, it sinks to the bottom of the container again because it is heavier than water. It should have a milky appearance and an oily consistency. It will usually cause the bicarb solution to start giving off little bubbles, or even some fizzing. This is the bicarb neutralizing the acid. Acid reacts with bicarb to make CO_2 gas. This is the reason a person belches after drinking some bicarb to treat heartburn.

The best way to handle this acid-neutralizing process is to rotate the container so that the nitro rolls around on the bottom of the container, exposing fresh surfaces to the bicarb solution. Swirling may also be attempted, as may gentle stirring with a wooden swizzle stick or plastic straw. A glass stirring rod should not be used because this could result in grinding friction between the stirrer and the bottom of the container, with the nitro trapped in between. This could have disastrous consequences.

If one has a sep. funnel, it is most convenient to do the neutralizing process in the sep funnel. This is because, once the nitro has been treated with the bicarb solution, it must once again be separated from the water. So if the bicarb treatment is done right in the sep. funnel, one avoids the hassle of transferring the liquids from one container to another, or using a turkey baster. When using a sep. funnel, it is best if the funnel is tilted at about a 45 degree angle to expose a greater surface area of the nitro to the bicarb solution.

However one chooses to do the bicarb treatment, one should spend at least a couple hours on this step. Letting it go overnight does not hurt. This does not mean that it must, or even should, be constantly attended, stirred, swirled, or fussed over. An occasional agitation IS enough to do the job well. More agitation and warmer water will get the job done more quickly, but why be in a hurry when one is having such fun?

At the end of the bicarb treatment, the nitro does not look any different from when it went into the process. It still has a milky appearance. This is caused by water trapped inside the nitro, and it should be removed before the nitro can finally be called pure. If it is left in the nitro, it could cause problems with detonating the nitro, or with further processing it into gelatin or dynamite.

The best way to remove the water from the nitro is to let it sit in contact with a saturated salt solution. This sucks up water from the nitro in the same way that drinking salt water dehydrates shipwrecked sailors. Saturated salt solution is water that is holding dissolved in it all the salt (regular table salt) that it can. The best way to make saturated salt solution is start with hot water in any container, and add salt to it until no more will dissolve in it. Good stirring or shaking is essential to getting the maximum amount of salt to dissolve in the water.

When the water cools down, the clear colored saturated salt solution (i.e., no crystals of solid salt floating around) is poured off of the excess salt sitting on the bottom of the container. This is put in a suitable container and the nitro is separated from the bicarb solution by use of a sep funnel or turkey baster as described earlier. Then the nitro is put in contact with the salt solution. There should about two volumes of salt solution to one volume of nitro. After they sit together in contact for a few hours, the salt will suck the water out of the nitro, resulting in a clear product. The product will be yellow if the glycerin was yellow to start with due to water removal processes. The yield of nitro is about 50 ml, the same volume of nitro as glycerin used in the manufacture. This process can be shortened if the bicarb solution is also saturated with salt. This will dehydrate the nitro at the same time it removes the acid from it.

This yield of nitro can be improved greatly by using the higher grades of acids. If one of the fuming acids is used, the amount of nitro obtained from the same sized batch is approximately doubled. This is due to the lesser amount of

water in the mixture, and the higher concentration of nitric acid. This method using the common concentrated acids was given first because these acids are the most easily available and cheapest. We shall now cover the small difference in processing when using the fuming acids versus the Plain concentrated acids.

Before moving on to production processes using the fuming acids, one more point should be made. Since the amount of nitro produced corresponds to the amount of glycerin used, a person may be led to believe that by using more glycerin in a batch of nitro, more product could be obtained. This is wrong. It does not work for reasons I shall explain.

Below is the molecular structure of glycerin:

```
 H
HC-OH
 |
HC-OH
 |
HC-OH
 H
```

Notice that it contains three alcohol groups (the OH sections of the molecule). Each one of these must react with nitric acid for the product to be nitro.

```
 H
HC-O-NO2
 |
HC-O-NO2    Nitro
 |
HC-O-NO2
 H
```

If the amount of nitric acid in the reaction mixture becomes stretched too thin. as will happen if too much glycerin is added to the mix, then by-products will begin to be formed where one or more of the alcohol groups of the glycerin has failed to react:

```
 H              H
HC-O-NO2     HC-OH
 |              |
HC-OH        HC-O-NO2  mono-nitroglycerins
 |              |
HC-OH        HC-OH
 H              H

 H              H
HC-OH        HC-O-NO2
 |              |
HC-O-NO2     HC-OH     di-nitroglycerins
 |              |
HC-O-NO2     HC-O-NO2
 H              H
```

So the use of more glycerin is self-defeating. These mono and diglycerins are explosive compounds, but are not so good as nitro itself. Some of these compounds will be formed in any nitro process, but the aim should be to keep their numbers as small as possible.

The natural next question is: How careful do I have to be at measuring out the ingredients to get good results? The answer is that, one can always use extra acids and still get good results. It's just that after a certain point, it gets wasteful. As for the glycerin' so long as the amount used is within 10% of the prescribed amount, no problems will be encountered. The familiar TV and movie image of chemistry requiring to-the-drop accuracy in measurement is true for doing analysis, but not usually for making chemicals.

Now let's move on to the variation of nitro production using fuming nitric acid and concentrated sulfuric acid. This process is no more difficult than the previous process, but fuming sulfuric acid is more expensive and more difficult to obtain. As was mentioned earlier, the yield of nitro is double the amount of glycerin used.

One starts this process with the same set-up as used in the first method. The tub of ice sits on the table top and the pail of water sits nearby for dumping out-of-control reactions into. The same type of reaction vessel (thin walled glass with a pouring lip) is set into the ice bath, and 100 ml of fuming nitric acid is measured out of the bottle or jug of fuming nitric acid that was previously cooled down in the freezer. To this is added 50 ml of glycerin. The addition should be slow, covering a period of about 10 or 15 minutes. It does not need to be as slow as in the first method because without the sulfuric acid in the mix, the reaction is not so vigorous. The cement mixer technique of stirring is

recommended here again because of its gentleness. The temperature should be watched carefully, and kept within the range given in the first method.

After all the glycerin has been added, the mixing should be continued for a few minutes. Then 200 ml of concentrated sulfuric acid (ice cold, fresh from the freezer) should be measured out. It is then added to the reaction mixture in small portions over a 10 minute period. The cement mixer technique of mixing should be continued during the addition of the sulfuric acid. The temperature of the brew should be watched during the addition to be sure it does not rise past the allowed limits.

There are two reasons why the temperature of the mixture will tend to rise during the addition of the concentrated sulfuric acid. First, there will be some water in the reaction mixture due to the fact that water is formed as a by-product along with the nitro. Recalling the original formula, three molecules of water are formed along with each molecule of nitro made. When concentrated sulfuric acid is diluted with water, a great deal of heat is given off. One can prove this to oneself by pouring some concentrated sulfuric acid into some water.

Secondly, when the sulfuric acid soaks up the water in the reaction mixtures, the reaction will shift into high gear again making more nitro. This will produce a good amount of heat. The reason why it shifts into high gear again is because when the water is removed, the equilibrium which was established between reactants and products is upset. The only way to reach the equilibrium point again is to form more nitro. See the equilibrium equation back on page 11 and prove it to yourself with some simple algebra.

When all the sulfuric acid has been added, the mixing should be continued in the ice bath for a few more minutes, then the reaction vessel can be taken out of the ice, and allowed to warm up into the 50's F. It should be held there for about 10 minutes with mixing, then the whole batch is poured into some water, just like in the first method. From here, processing into pure nitro is handled exactly as in the first method. The only difference is that 100 ml of nitro results instead of 50 ml.

Lastly, we come to the final variation on nitro production. This is when the acids used are concentrated nitric acid (i.e., 70%) and fuming sulfuric acid. There are a couple complications to this method. First of all, there are two common grades of fuming sulfuric acid, one contains 20% SO_3 (oleum) and the other contains 40% SO_3. So separate directions will have to be supplied for each grade of fuming sulfuric acid. Secondly, fuming sulfuric acid reacts pretty vigorously when it comes in contact with water, so it must be mixed up front with the nitric acid, rather than added last as in the previous method. If it was added last, the heat it would produce would send the reaction over the limit. These complications are easily overcome.

So to start, the same setup is used as in the previous batches. Then into the reaction vessel nestled into the ice bath is put 100 ml of concentrated nitric acid (70%). This is ice cold stuff fresh from the freezer. Then to this is added slowly with good stirring 115 ml of 40% SO_3 fuming sulfuric acid, or 200 ml of 20% SO_3 fuming sulfuric acid. This fuming acid will react vigorously when it hits the nitric acid because the nitric acid is 30% water. It may even splatter, so the opening of the vessel should point away from the experimenter. It is best if the fuming acid is allowed to run down the walls of the reaction vessel into the nitric acid. Stirring is called for during this operation to keep the reaction dispersed.

When all the fuming acid is added to the nitric acid, they must be mixed thoroughly, then allowed to cool down in the ice bath until the temperature of the mixed acids is under 40ø F. Then it is time to add the glycerin. 50 ml of cold glycerin is measured out and added in 5 ml portions over a 20 minute period, just as in the first method. The cement mixer stirring technique is once again king. When all of it has been added, mixing is continued for a few minutes, then the brew is taken out of the ice bath and allowed to warm into the 50's F for a while as before. Then it is poured into water, and purification to pure nitro is done exactly as in the previous methods. Yield of product is a little over 100 ml if 40% oleum is used and about 100 ml if 20% oleum is used.

So, as one can see, making nitro is not an especially difficult procedure, if one has at hand the required chemicals. Equally easy is gearing this process up for turning out substantial quantities of nitro.

As was alluded to earlier, larger batches of nitro have dangers inherent to their production that the smaller batches do not. For instance, a runaway big batch may blow up rather than just make poisonous gases. So the aim of the Home Workshop experimenter should be to organize his production process to take advantage of the dead time in the purification of the nitro to start new batches, rather than to try to push out monster batches.

The longest stretch of dead time in the production process is the acid neutralization with Arm & Hammer bicarb. This several hour long wait would be a tremendous bottleneck in the production process if one would just sit around and wait for it to get done. On the other hand, if the waiting time for the acid to get neutralized is spent starting another batch, no bottleneck is experienced. All one would have to do is clean up and dry the reaction vessel, run another batch, and then put it through the purification process. In this way, one would have several batches running at once, all at different stages of the production and purification process.

Another section of the purification method that involves a lot of just sitting around and waiting is when the nitro is put in contact with saturated salt solution to remove the water from it.
This dead time can be profitably used in the same manner. It is obvious that a fair number of containers of crude nitro will be employed in such an operation. They should all be clearly labelled so that mix ups are avoided.

Organization and logical thinking are indispensable in this situation, but using this method of organization, production levels in excess of a pint a day of pure nitro are easily obtainable.

Production can be further increased if there are two people to man the production line. The most fruitful way to divide labours in such a situation is for one person to devote himself to running the synthesis reaction between the glycerin, nitric acid and sulfuric acid, and for the other person to man the purification operation. With this division of labours, production can easily exceed a quart per day without resorting to larger batch sizes.

When the nitro has reached the end of the purification process after the saturated salt treatment, a perfect place to store the nitro is in the freezer. The colder the nitro is, the more stable it is. Cold nitro is pretty difficult to detonate, and frozen nitro is next to impossible to detonate. A glass container is wonderful for this purpose. As subsequent batches get finished, the product can simply be poured in with the rest of the nitro in the freezer until the container is filled.

When the desired amount of product has accumulated, or when the freezer is full, a decision must be made. That is whether to keep and use the nitro in its pure liquid form, or to convert it to blasting gelatin, or even to ruin it by mixing it into a dynamite. In making this decision, the full range of factors have to be weighed. For instance, one has to consider what is going to be used as a detonator for the explosive. If one has access to blasting caps, or can improvise a low-powered cap, gelatin is the nitro-based explosive of choice. Gelatin, when fresh, retains the full power of the nitro, and yet is a good deal safer than the pure nitro.

On the other hand, when all one can procure for detonating the explosive is a strong firecracker, or if the nitro is going to be used for aerial bombing, then pure liquid nitro may be the way to go. More on this when detonation systems are discussed later.

If gelatin is the chosen route, one can take heart from the fact that gelatin is very easy to make from nitro, and that so long as it is fresh, it packs an amazing punch. What is meant by "fresh" is that the little air bubbles that get whipped into it in the process of making it are still visible. Once they disappear, half the power is lost because the detonation rate goes down.

Now for how gelatin is made. You may be surprised after reading this and say, "What? There's got to be more!" Really, there is just nothing to the process. First of all, a suitable mixing bowl and stirrer combination is rounded up. A really good combination is a stainless steel bowl and a rubber spatula (small size). Also workable is china with wooden stirrers. What must be avoided at all costs is a hard bowl with a hard stirrer, like a glass stirrer on metal or china bowl. The grinding friction during the mixing could easily set off the gelatin. A combination that generates static electricity such as plastic on rubber must also be avoided because a little spark could set the guncotton to burning.

Now into the stirring bowl is put 93 grams (58 ml) of nitro. Then 7 or 8 grams of nitrocellulose (guncotton: see methyl nitrate section for a discussion of guncotton) is stirred into the nitro. If the nitro is cold, adding an ml or two of acetone (available off the shelf at the local hardware store, check in the paint section) to the mix will help a lot in forming a good even mixture. The use of acetone can be avoided if the nitro is warmed gently by setting the bowl in warm water before adding the guncotton.

When the guncotton is all mixed in, a white jelly will result. The more guncotton that is added, the stiffer the jelly will be, the harder it will be to detonate, and the weaker it will be. Gelatin with 7% guncotton (i.e., 7 grams guncotton to 93 grams nitro) when fresh can be set off with a #1 blasting cap (the weakest) and with a #4 when it has set long enough that the gelatin has turned from white to dear yellow. 9% guncotton in the gelatin requires a #3 or 4 cap when fresh, and stronger caps when aged.

The gelatin formed in the mixing bowl can be removed using the spatula and stored in ziplock bags until ready for use. Larger batches of gelatin can be easily made, so lone as care is taken to ensure that the mixing is complete and even. A uniform product should result.

NITROGLYCOL

Nitroglycol (or ethylene glycol dinitrate) is an excellent alternative to nitroglycerin. It combines almost all of the good points of nitro with a few further unique advantages of its own. Its easy manufacture (just like nitro!) and high power make nitroglycol an attractive choice for any job requiring shattering explosive power.
One could go on endlessly expounding the points in favour of recommending nitroglycol, but there is no need to go into such detail. By knowing the major advantages of nitroglycol, the reader will be able to make his own informed choices.

The first and foremost advantage of nitroglycol is that it is less sensitive than nitro. It requires three or four times the force to set it off. This should be very reassuring to all the timid hearts contemplating entering the explosives manufacture field. On the serious side, this difference in sensitivity becomes very important when large amounts of the pure liquid explosive are being handled.

Another good point about nitroglycol is that it has better storage qualities than nitro does. Nitroglycol is much better able to withstand a sloppy purification job. This is because traces of acid left in the product will not bring nitroglycol to as rapid a ruin as nitro. This should not be interpreted as an endorsement of sloppy purification methods. It just means that with nitroglycol, there is more room for error.

For arctic blasting operations, or just the dead of winter, nitroglycol has the advantage of being explodable at temperatures which would freeze nitro into a harmless solid mass. This is because nitroglycol freezes at a much lower temperature than nitro does. Nitroglycol is also much runnier than nitro is. This low temperature explodability can also be viewed as a disadvantage since freezer storage can't be considered as safe as with nitro.

Finally, nitroglycol is very easy to use. It can be used in all the ways that nitro can. It forms gelatins with guncotton much more easily than nitro does. No warming or addition of acetone is required to get a gel to form.

Nitroglycol is not without its weak points, however. One bad thing about nitroglycol is that exposure to it brings on headaches much easier than does nitro. Contact with the skin can bring them on, as can breathing the vapours. When working with the substance, one should be wearing rubber gloves. Surgical type gloves are best because they do not interfere with manual dexterity like the heavier gloves do.

This is a minor drawback. A much more serious difficulty is that the starting material for making nitroglycol, ethylene glycol, is not available directly off store shelves. This complication would be very frustrating were it not for a couple of ways around it.

The first and simplest way around this problem is to order the chemical from the various chemical supply outlets. Since ethylene glycol has no major drug production usages, major hassles should not be expected. A reasonable price is about $30 per gallon. Any orders for this substance should be placed at a different outlet than the source of the acids.

In many cases, it is the wisest route to avoid chemical purchases when possible. The paper trail they can leave is unsettling for covert operations. Luckily for these situations, there is an easy and cheap alternative source of ethylene glycol: antifreeze.

Other publications have suggested making ethylene glycol by warming ethanol with sulfuric acid to convert the ethanol to ethylene gas and then passing this gas through a bed of coke impregnated with phosphoric acid, and then feeding this product into nitroglycol production. This route is silly for the Home Workshop Explosives manufacturer. It is much too involved and subject to failure for workshop use. This method would be much more at home in a large factory manned by a staff of experienced chemists. The motto to follow is "keep it simple." Anything that can go wrong is likely to, over the long haul.

A quick lesson in antifreeze formulations will well serve the experimenter interested in obtaining usable ethylene glycol from it. Antifreeze has as its major ingredient ethylene glycol (unless you live in Russia where vodka is popular). In addition to the ethylene glycol there is an additive package in the antifreeze. This package varies with the brand of antifreeze, but the major additive is triethanolamine. This ingredient is universally used to inhibit rust formation in the radiator. The better brands will have in addition to triethanolamine, substances intended to plug small leaks, and other additives as well to extend rust protection to aluminium radiators and so on. These more complex additive packages are bad news to the Home Workshop manufacturer, because the lengthening list of additives make the job of getting pure ethylene glycol from antifreeze very troublesome.

To avoid these difficulties, the cheapest brand of antifreeze is sought out. Generic antifreeze is ideal. It is likely to contain only triethanolamine as an additive. Now the manufacturer is faced with only the problem of removing the triethanolamine from the mixture. The triethanolamine must be removed because it will not stand up to the mixed acids used to convert the ethylene glycol to nitroglycol. Triethanolamine will break down under those conditions and cause the formation of the dreaded red gas, NO_2, if it is left in the mixture.

To obtain pure ethylene glycol from the cheap antifreeze, distillation is used. This is the way to go because ethylene glycol boils at 197ø C, while triethanolamine boils at 335ø C. A very good separation of the two substances can be obtained by this method without resorting to fractionating columns.

The best and simplest way to do this distillation is if the Home Workshop experimenter has invested in the distillation kit described in the nitric acid section. Such glassware comes in handy again and again for the serious experimenter. The only bad thing I can say about owning such equipment is that it is expensive, costing several hundred dollars. Narco swine surveillance centers on chemicals, not equipment.

Using the chemical glassware, obtaining pure ethylene glycol can proceed very simply. See the drawing on page 59. The larger distilling round bottom flask sits directly on the electric hotplate. The so-called "fifth burner" unit bought at the local hardware store for $20 is perfect for this job. It should have an infinite control knob to regulate the amount of heat it gives off. The antifreeze is put in this flask, filling it about half or 2/3 full. A few pea sized chunks broken off of a Dr. Scholl's pumice footstone should also be put in this flask to ensure a smooth boil during the distillation.

The way the distillation is done is as follows: First the flask sitting upon the heater element is filled about 1/2 or 2/3 full of antifreeze. The heat is applied from the fifth burner unit until some boiling begins in the heated flask. These vapours will work their way up through the neck of the flask, and droplets will begin to appear on the thermometer, and in the area of the stillhead where it makes the turn to deliver into the condenser. When these droplets appear, which is soon after boiling begins in the heated flask, water flow should begin through the condenser. This will cool off the vapours and turn them back into a liquid. These condensed vapours will flow down into the other flask where they can be collected when all the product has arrived. The rate of boiling in the heated flask should be controlled by varying the amount of heat applied to it so that a slow trickle of about one drop per second appears in the other flask

Let's now focus in on the stillhead, because it plays such a crucial role in the distillation.

The thermometer should extend precisely to the position shown in the drawing. Any other placement will give false temperature readings. Pushed down lower, it will give false readings on the high side. Placed higher up, it will read too cold.

The second thing which can be done with the stillhead is that its lower end can be packed. This will result in a much purer product because a little bit of packing will act as a fractionating column. To do this, a glass bottle is smashed on a cement floor, and the resulting pieces are picked over to find a few small enough to fit in through the top, yet big enough to keep from falling through the bottom. A few pieces of glass are enough to do the job. Alternatively, a little bit of a stainless steel scrub pad can be pulled into this area.

When distilling the antifreeze, the temperature showing on the thermometer should be watched closely. Since ethylene glycol boils at 197ø C, the liquid appearing in the collecting flask should only be saved when the thermometer indicates that the vapours entering the condenser are close to that temperature. To clear up any misunderstanding, let's go through a typical distillation.

When the vapours first reach the thermometer, the temperature shown on the thermometer will rise quickly. It is unlikely, however, to scoot up immediately to 197ø C. First the ingredients in the antifreeze with lower boiling points will distill off. For example, if it is really cheap antifreeze, some water will boil off at 100ø C. None of this lower boiling material should be saved. It should be collected in a separate container and thrown away. Only when the temperature shown on the thermometer gets into the 190ø C range, should the liquid appearing in the collecting flask be considered to be ethylene glycol.

When the ethylene glycol begins appearing, the heat should be turned down some on the fifth burner unit to avoid superheating the antifreeze and giving false high readings on the thermometer. Collection of ethylene glycol should continue until the temperature shown on the thermometer goes above 205ø C. About 2/3 of the antifreeze put in the distilling flask will be collected as pure ethylene glycol in this manner. When the temperature goes above 205ø C, the heat should first be turned down further on the fifth burner unit to see if slower distilling will produce more ethylene glycol. If the temperature persists above 205ø C, then all the ethylene glycol has been collected. When this point has been reached, the heat should be turned off, ethylene glycol should be poured into a clean, dry glass container and the top tightly closed. It must be closed because pure ethylene glycol will soak up water from the air and dilute itself. The presence of water in the ethylene glycol will have the bad effects on yield of product already discussed in the previous sections of this book. The slop antifreeze material left in the distilling flask should be poured down a drain when it cools off. Then another run of fresh antifreeze may be distilled if desired.

It is not necessary to possess chemistry glassware to distill antifreeze. The stuff is not corrosive, or otherwise particularly dangerous (so long as it is not drunk, or the hot fumes sniffed!) so a homemade still can be constructed and used for the process with good results so long as care is taken in its construction and use.

An antifreeze distillation using this setup will proceed in the same manner as described for the chemical glassware version, with the difference that it will not be possible to as closely monitor the process. With some practice, and using the same brand of antifreeze, it should be possible to get as good results using this apparatus as with the chemical glassware.

A few words are in order on the construction of this still, as there are a few trouble spots. First and most important is the cork or rubber stopper in the metal can. It must fit tightly into the opening in the metal can so that the vapours are forced up the tubing. Leakage here can result in fluid running down the side of the can, posing a fire hazard. The holes in the stopper must be exactly the right size to hold the tubing and the thermometer. Ragged edges on these holes are not acceptable, as the resulting leakage could pose the same fire hazards. Cork borers are available which do a good job of punching clean holes through corks. In their absence, a very careful job must be done in

making these holes. Loose spaces and ragged edges can be mended by use of silicone bathtub caulk just prior to inserting the tubing and thermometer.

The coiled portion of the metal tubing can be made by wrapping the copper tubing around a sturdy round object. Care must be taken to avoid kinking the tubing shut during this process.

Finally, some support must be provided for the copper tubing so that its weight does not cause the metal can to tip over, or cause damage to the cork. Any type of support is OK for this purpose, so long as it does the job. This requirement for support is not unique to the homemade still, as the chemical glassware set-ups also need clamping into place or other measures to hold the pieces together.

Doing a distillation using this homemade still works almost exactly the same as with chemical glassware. There are only two areas where the method differs. The first difference is that the thermometer inserted into the top of the can will measure the temperature of the vapours there, not at the top of the metal tubing where it begins its descent. This means that temperature readings on the thermometer are likely to be falsely high. With some practice and always using the same brand of antifreeze, this can be corrected for on an empirical basis.

The second and potentially more troublesome problem has to do with the design of the coiled condenser portion of the copper tubing. As shown, it relies solely on air cooling for the vapours to give off their heat and become liquids once again. This will be sufficient during cold weather in the workshop, if the rate of distilling is slow enough. Otherwise some plastic tubing can be wrapped around the coiled portion of the metal tubing, and a flow of water run through the plastic tubing. The bottle into which the product drips can also be cooled off by packing it in ice. Under no conditions is it acceptable for steam to be escaping from the end of the metal tubing. Enough cold must be applied to the condenser to turn the vapours back into liquids.

MAKING NITROGLYCOL

With the ethylene glycol in hand, one is ready to turn it into nitroglycol. This process is very easy and almost identical to the procedure used to turn glycerin into nitro. To illustrate just how similar this process is to the nitro manufacture process, see the formula below:

```
  Nitric + Sulfuric
OH-CH2=CH2-OH => NO2-O-CH2=CH2-O-NO2
      Acids
Ethylene Glycol          Nitroglycol
```

Compare this to the reaction for making nitro. Here the alcohol is ethylene glycol. For nitro, the starting alcohol is glycerin. The only difference in this reaction is that ethylene glycol is one carbon atom shorter. As far as the practical detail of making the two substances go, there is very little difference. All three of the variations on the nitro making process work for making nitroglycol, just by substituting ethylene glycol for glycerin. The same amounts of all ingredients are used.

There are only two small details where the production of nitroglycol differs from nitro. The first difference is that nitroglycol is more prone to form the red gas, NO_2 during its manufacture than is nitro. Because of this tendency, the ethylene glycol should be dripped directly into the acids rather than allowing the stuff to run down the sides of the glass into the acids. More attention should also be paid to getting the ethylene glycol promptly stirred into the acid mixture. It may also prove to be more fussy about the quality of the nitric acid used in the process than is the case with nitro.

The second detail in which nitroglycol manufacture differs from nitro production is the greater ease with which nitroglycol dissolves in water. As a result of this, less water must be used in washing up the nitroglycol than was the case for nitro. If the full amount of water was used, excessive amounts of product would be lost by getting dissolved in the wash water, and thrown away. To minimize these losses, the final wash with saturated salt solution should definitely be combined with the bicarb wash. This is done by the method described in the nitro chapter. The bicarb solution has salt added to it until it can't dissolve the added salt anymore. Then this wash water is used on the crude nitroglycol.

NITROMANNITOL

Nitromannitol (aka nitromannite) is the third most difficult to make of the good high explosives. Other factors being equal, then nitromannitol should be the third choice for the Home Workshop Explosive manufacturer when setting up shop.

The production of nitromannitol is relatively easy because the starting material for its manufacture, mannitol (mannite), is easily available at a moderate price from headshops. It is sold wherever fine dealing supplies are sold because mannitol is a popular cut for cocaine. The person behind the counter is likely to give you a knowing wink when making the transaction, but have no idea of its usefulness in explosive manufacture. If the heat has succeeded in driving all the headshops in your area out of business, High Times may carry ads for mail-order mannitol. This is not certain, however, as their snotty "My dope is better than your dope" attitude may cause them to refuse to carry such ads in the future.

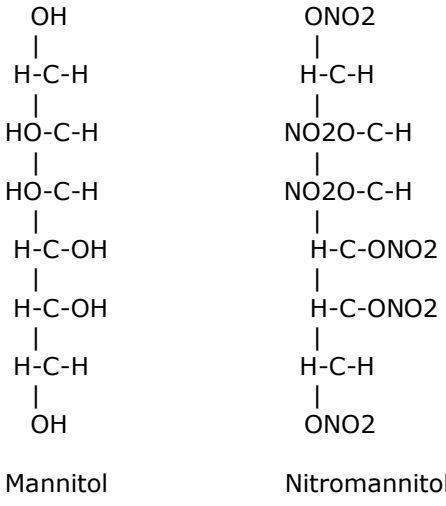
The production of nitromannitol is not without its unique pitfalls, which combine to make the manufacture of nitromannite about twice as difficult as making nitro or nitroglycol. Foremost of these difficulties is the sensitivity and poor keeping properties of nitromannite. Under the best of conditions, it is about as sensitive as nitro, but if it is not completely freed of all traces of acid during the purification process, it will rapidly become too dangerous to handle.

Removing these last traces of acid is difficult because nitromannite is a solid, and so can lock the acid inside its crystals, beyond the reach of wash water or bicarb. To get at these locked-up acid residues a new chemical technique must be introduced here. This chemical technique is called recrystallization. Recrystallization involves dissolving the nitromannite in a solvent, like alcohol, and then allowing the crystals to reform from this solvent. This results in the garbage which was formerly locked up inside the crude crystals being released into the solvent. The new crystals which form are largely free of their former pollutants. This may sound complicated but it is surprisingly easy to do. Complete, detailed instructions will be given in the text.

The other new technique which must be mastered to produce nitromannite is vacuum filtration. This technique is needed because nitromannite is a solid with very fine fluffy needle like crystals. It has to be separated from its reaction mixture or recrystallization liquors by filtration. These long, fine needle crystals can only be filtered properly by having a vacuum force the flow of material through the filter. Without this, filtering the batches could take hours, and the results would be poor. This technique is very simple and can be improvised using household equipment. It too will be explained in detail when the time comes.

Making nitromannite proceeds in a remarkably similar manner to the manufacture of other explosives covered to this point. It is a nitric ester like the rest, and so is made by reacting an alcohol, in this case mannite, with nitric acid. See the reaction on next page.

As can be seen from the drawings, mannitol has 6 alcohol groups (OHs) which react with nitric acid to form nitric ester groups (ONO2s). Other than the large number of alcohol groups mannite is almost identical to glycerin or ethylene glycol.



Mannitol Nitromannitol

This large number of alcohol groups has an important practical effect upon what strength acid may be used in its manufacture. Referring back to the equilibrium equation given in the nitric ester section for nitroglycerin, the same equation can be written for nitromannite simply by raising the nitric acid terms and the water terms to the 6th power instead of the third power. This means that it is crucial in making nitromannite that the strongest possible nitric acid is used, and as a natural consequence (since the diluent for nitric acid is water) that the amount of water in the mixture be held to an absolute minimum. For this reason, only fuming nitric acid (at least 90% strength, density 1.5) can be used to make nitromannite. This fuming nitric acid must either be obtained through commercial sources, or made according to the directions in the nitric acid section. The absolute need for the high strength acid is one of the roadblocks to putting nitromannite into production in the workshop.

To set up for making nitromannite, one begins with the same equipment as used earlier. The glass reaction vessel, pail of water and tub filled with ice are all needed. The important difference is that the temperature of the reaction must be held to 0øC (32øF) or below, so the ice must be a good deal colder than usual to keep the reaction at such a cold temperature. The way this is done is to mix the ice with about half its volume of table salt. This will drive its temperature down to around -20øC (0øF) which is cold enough to do the job against the heat given off in the reaction. An alternative is to pour a bottle of rubbing alcohol onto the ice and mix it around. This will also lower the temperature of the ice greatly.

Then into the reaction vessel, nestled deeply into the ice bath, 100 grams of fuming nitric acid (66 ml) is poured into the reaction vessel. Of course, the nitric acid should come directly from the freezer so that the ice does not waste itself cooling down the nitric acid. To this nitric acid, 20 grams of mannitol is added a little bit at a time. Between each addition of mannitol, the contents of the reaction vessel should be mixed by swirling. It is best to move the whole tub when swirling the reaction so that close contact of the reaction vessel with the ice bath is not broken. A thermometer should be in the reaction at all times, and it should be watched so that the temperature does not go above 0øC. If it does, the reddish brown fumes of NO_2 are likely to follow. In that event, the batch gets dumped into the pail of water. So long as the addition of mannitol is slow, and the temperature is watched, this event is unlikely. The addition of the mannite should take 20 or 25 minutes.

The mannite should dissolve quickly as it is added to the nitric and mixed in. To ensure rapid dissolving and quick reaction, the mannitol should be finely powdered before it is used. Oftentimes, mannite is sold in head shops in quarter-ounce pressed blocks. These blocks will have to be mashed with a fork to break them up.

After all the mannitol has been added, and the last of it has dissolved into the nitric acid, it is time to finish up the reaction. This is done by adding concentrated sulfuric acid to the reaction mixture. This has the effect of sucking up whatever water is present in the mixture, thereby forcing the reaction to completion, and also causing the nitromannitol which has been produced to come out of solution and form crystals which can then be filtered out.

To finish off the reaction, 200 grams (110 ml) of concentrated sulfuric acid is measured out. The sulfuric acid should be ice cold, fresh from the freezer. It is then added a drop or two at a time with an eyedropper (glass stem!) to the reaction mixture. Between each drop or two, the acid should be mixed in by mixing with the eyedropper or the thermometer. The temperature should be watched closely during the addition of the sulfuric acid to make sure the temperature does not go above 0ø C. As the temperature nears 0ø C, the addition of sulfuric acid should be stopped until the mixture cools off again.

As the sulfuric acid is added, crystals of nitromannite will appear, slowly at first, and then quite rapidly. By the time half of the acid is added, the mixture will be getting pretty thick from the large mass of crystals formed. As the mixture gets thicker, it will be harder to mix in completely the added acid, so extra effort must be put into ensuring an even mix as the last of the acid is added. Care must be exercised in using the thermometer as a mixer here so that it is not banging the sides of the glass reaction vessel, or grinding the bottom. This grinding could provide the friction to set the whole batch off.

Finally, when all the acid is added, a mixture with the appearance of a good heavy slush will be formed. Now the challenging part of the operation begins. The crystals of nitromannite must be filtered out of the highly corrosive nitric and sulfuric acid mixture in which they are floating, quickly enough that they do not rise above the red gas-forming temperature before they are rinsed with water and thereby rendered more stable. This operation is complicated by the fact that the nitric and sulfuric acid mixture in which they are floating will fairly quickly react with filter paper to form guncotton. This will cause most of the filter paper to dissolve, and ruin its effectiveness at catching the crystals.

There are a few ways around this problem. The most important part of the solution is to get the filtering done as quickly as possible. At least the first part of the filtering must be done very quickly, the filtering out of the crystals from the acid reaction mixture. Later, when it is rinsed with water and rendered less corrosive, a more leisurely pace can be taken. To make this filtration a quick process, there is only one way to go. That is to have a vacuum pulling from the underside of the filter, dragging the liquids through as fast as possible. Actually, this would be the only way to go even if the acids didn't eat filter papers. Packing the stuff inside a large coffee filter and wringing it out by hand is madness. Allowing it to sit in a funnel and drip through a filter is silly as well because the combination of fluffy crystals and viscous acids (concentrated sulfuric acid is about as thick as light motor oil) will make this take so long that it is unreasonable, even if one did not have to worry about the temperature of the mixture rising above 0øC. Genuine chem lab experience is what separates this book from many pretenders out there, most of which merely feature reprints from scholarly works which assume the reader has a high level of chemical skills and can solve these problems for himself without giving all the details. Place your wild-eyed trust in Uncle Fester. You won't be disappointed.

Let us first see how this rapid filtration problem would be handled using chem lab equipment before looking at ways to improvise for the same effect. To begin with, a source of vacuum would be dose at hand on the lab bench, such as an aspirator. A water bed pump would serve the same purpose just as well since a really good vacuum isn't needed. From this vacuum source, a vacuum hose similar to an automotive vacuum hose would be led to the glassware set-up.

The filtering flask has a side tube to which a vacuum hose attaches, producing a vacuum inside the flask. This vacuum serves to rapidly pull fluids that are poured into the Buchner funnel down into the flask. This Buchner funnel normally has a flat bottom inside it at about the level shown by the dotted line in the drawing. The flat bottom is perforated

with many holes, and this is the place where a filter paper is laid, so crystals are stopped at the filter paper. The fluid proceeds through down into the filtering flask, and the crystals pile up in the Buchner funnel above the filter paper.

Now using this chem lab equipment, the problem of filtering would either be handled by piling up several filter papers, soaking them wet and hoping that the wetness would protect them through a rapid filtering (bad solution), or by using something other than filter paper to catch the crystals, something which would stand up to the acids without being eaten away.

A great alternative to ordinary filter paper for this job is asbestos paper. This useful material used to be very easy to get at hardware stores, but has since been outlawed, along with virtually anything else made of asbestos, in the U.S. It has since been replaced with a woven glass sheet like material which works almost as well as asbestos paper. Its only drawback is that it tends to be thick and stiff, and so is not so easy to work with. To use this woven glass sheet as a filter, one would only have to carefully cut a piece out of it with a scissors that exactly fits the bottom of the Buchner funnel. Once in place, the slush like nitromannite could simply be poured through it with the vacuum applied to the filtering flask, and all the acids would rapidly be pulled through to the filtering flask and the crystals of nitromannite would pile up in the Buchner funnel.

To modify this process for using common household materials, one should first look for a replacement for the Buchner funnel. This is easily enough done by heading to the hardware store and getting a plastic funnel with a long narrow stem and a plastic screen in the bottom. This funnel very closely approximates a Buchner funnel. Cost: about $2.

Next, to replace the filtering flask, one can use a Mickey's Big Mouth malt liquor bottle. This rather sharp-tasting brew is made by the Old Style Brewery. Cost: nothing, if you like beer.

Finally, to replace the one-hole stopper, a two-hole stopper of the correct size is needed to fit in the top of a Mickey's Big Mouth bottle. If easy access is not available to an assortment of different size stoppers so that you can choose the right one, the Edmund Scientific catalogue usually has quite a variety to choose from with no suspicion involved. This improvised equipment can be set up as shown on following page.

This is basically the same set-up as with the chem lab glassware except that the vacuum hose does not attach to the Mickey's Big Mouth bottle since it has no side tube to put a vacuum hose on. Instead the vacuum hose attaches to a stiff plastic or wooden tube (no copper allowed! improvise!!) inserted through one of the holes in the two hole stopper.

This is the source of the vacuum inside the Mickey's Big Mouth bottle. The stem of the plastic funnel goes through the other hole in the two hole stopper. Greasing the stem of the funnel with vaseline will greatly ease its passage through the two hole stopper.

Next a section of the woven glass material mentioned earlier must be picked up at the hardware store. A section should be carefully cut out of it so that it exactly covers the plastic screen in the bottom of the funnel. Good fit is essential here as any loose spots or uncovered areas or points of buckling of the filter will be places where nitromannite crystals can escape being caught, and end up going through with the acids. It may help to wet the filter and push it into place just prior to filtering the slush.

The procedure to use in filtering the material is this: First of all, the filtering apparatus is set up as shown in the pictures. Then the filter is put into place and wetted, then pushed down securely into place, making sure that the filter lays flat with no buckles or loose edges. Now the vacuum source is turned on, and the vacuum hose attached to the glassware.

Next, the slush like nitromannite mixture is poured into the funnel. Not all of it should be poured in at once, of course, because this is likely to overflow the funnel unless the funnel is very large. At first, the acids will quickly rush through the filter, but as a bed of nitromannite builds up in the funnel, it will slow up, and a greater vacuum will accumulate in the glassware to pull it through. The first rush of liquid through the filter should be watched closely to make sure that no significant amount of nitromannite crystals is coming through. If there is, this indicates that the filter is poorly cut or placed. It should get better as a bed of nitromannite builds up above it, but in any case the material which passed through will have to be refiltered separately at the end of the process.

The filtration will proceed rapidly if a reasonable vacuum is applied to pull the liquids through. The bed of nitromannite in the funnel (generic term: filter cake) will rapidly shrink as the acids are filtered out. If the right size funnel is chosen, all of the batch of nitromannite will fit in the funnel. As the last of the acids in the slush are filtered out, cracks will appear in the filter cake. Now it is time to move on to the next step, rinsing off the crystals.

The aim of all the purification steps in nitromannitol processing is to remove the residual traces of acids from its manufacture. The first step in this process is to rinse the crystals with water. To do this, simply pour cold water into the funnel on top of the nitromannite. It is convenient to rinse out the crystals clinging to the walls of the reaction vessel at the same time as the first rinse. This gets them in with the rest of the product with a minimum of hassles. This first rinse of cold water should be followed with a couple more, each one amounting to a couple hundred mls of water. For these last couple of rinses, it is good to turn off the vacuum (remove vacuum hose first to avoid water backup into acids) and just let the water move through the crystals slowly. Stir around the bed with a toothpick to avoid channel formation.

The next rinse for the crystals is a weak bicarb solution. This will get most of the remaining acids. A few grams of Arm & Hammer in a couple hundred ml of water will serve the purpose nicely. It is poured through the filter cake in the same manner as the water rinses were. Finally, another rinse of cold water is poured through the crystals. When the vacuum pulls most of the water out of the filter cake, it is time to move on to the recrystallization of the product.

A glass, china, or plastic plate is a convenient place to dump the product out of the funnel onto. The best way to do this is to carefully (after the vacuum is removed) pull the funnel and the stopper out of the glassware, and then tip the funnel upside down an inch or so above the surface of the plate. Some gentle tapping with the hand on the side of the funnel should be enough to make the filter cake fall out of the funnel. Avoid the temptation to bang the funnel on the surface of the plate to dislodge the filter cake. When it falls out, a plastic spoon can be used to scrape out whatever dings to the funnel. The filter should be picked out of the cake by hand.

To recrystallize, a heat resistant glass container must be obtained. A good choice is a one-cup size pyrex measuring cup. The crystals are put into the cup. Next some alcohol is added to the cup to dissolve the crystals. The best choice for alcohol is 190 proof grain alcohol. The next best choice is 91% isopropyl alcohol, available at the corner drug store right off the shelf. Unsuitable for use are denatured ethyl alcohol (because acid may have been used to denature it) and 70% isopropyl alcohol (because of too much water in the alcohol). Methyl alcohol (wood alcohol) is not a good choice because it dissolves the crystals too well, and so causes loss of product, and a poorer job of removing the last traces of acids.

The procedure used to dissolve the crystals is as follows: First, 100 ml of alcohol is added to the crystals. They will not dissolve noticeably in the alcohol because it is cold. A pan of water is boiled, and when it boils it is brought over to the table in the workshop and the measuring cup is put into the pan of hot water. This work should not be done on the stove because when the alcohol heats up, it will give off fumes which could be ignited on the stove. The hot water will quickly warm up the alcohol in the measuring cup, and it will start to dissolve the crystals of nitromannitol. The alcohol should be stirred to help with the dissolving of the nitromannitol. A perfect tool for this job is a plastic coffee stirrer from McDonald's. After a few minutes, the water will have cooled off to the point where a fresh pan of boiling water should be used for the heating of the alcohol. Soon after putting the measuring cup in the second pan of hot water, all the crystals of nitromannitol should be dissolved. If they have not all gone into solution, then some more alcohol should be added to the measuring cup to dissolve them. Since the added alcohol is cold, a third pan of hot water will then also be needed to bring the temperature of the alcohol up to the required level to dissolve the nitromannite.

When all the crystals have dissolved, the cup should then be removed from the pan of hot water and allowed to cool. As the alcohol cools, crystals of nitromannite will reappear. This is because cold alcohol does not dissolve nitromannite very well, so they are forced to come out of solution. These recrystallized crystals of nitromannitol are much purer than the crude material that was started with because the acids that were formerly locked inside the crude crystals remain in the alcohol.

The colder the alcohol gets the less nitromannite it will dissolve so the measuring cup should find its way to a freezer, or be packed in ice. This will get the largest amount of product out in this step. The nitromannitol should be white and the crystals should be long and needle shaped.

Now the pure product can be collected. To do this, the same filtering setup described earlier is used, except that a regular coffee filter can now be used, since there are no strong acids left for it to be dissolved by. All pieces, of course, must be clean and dry to avoid contaminating the product.

The alcohol which filters through contains some more nitromannite in it. To get this second crop of crystals out of it, the alcohol is simply poured back in the measuring cup (or beaker, which could easily be ordered from the Edmund Scientific catalogue) and heating is recommenced. The heating must be stronger this time. If grain alcohol (190 proof ethyl alcohol) was used, the mixture should be brought to a boil. This can be done safely for small batches like this one on an electric stove top by setting the cup in a pan of hot water and bringing the water to a boil with the overhead fan running to suck up the alcohol fumes. If isopropyl alcohol was used, no attempt to bring the alcohol to boiling should be made because isopropyl alcohol boils at the same temperature that the crystals melt at. This high temperature will prevent their formation during the crucial water-adding step to follow. For the isopropyl alcohol, just heat the alcohol up in the boiling water.

When the grain alcohol starts to boil, or when the isopropyl alcohol gets hot, water should be dripped into the alcohol. In the case of isopropyl alcohol, it will also be necessary to stir the water into the mix. The mixture should be watched closely during this addition. When the mixture starts taking on a milky color, stop adding water. This means when the whole solution gets milky, not just little areas, because local milkiness in the area where the drops of water land will be seen soon after starting the water addition. This milkiness is termed "turbidity."

Other publications have erred seriously on this point. They defined turbidity as a churning, as if it was going to get up and do a dance for them. It makes one wonder.

When this milkiness is seen, the heat should be removed, and the mix allowed to cool. Crystals of nitromannite will soon be evident. The milkiness was caused by small crystals of nitromannite forming in the solution. They formed because water is an even worse solvent for nitromannite than is cold alcohol. As the alcohol became progressively more watery, its ability to dissolve nitromannite went from bad to worse.

When this mix gets cold in the freezer or by packing in ice, the last of the nitromannitol crystals will have formed. They should be filtered out like the rest, and the alcohol which filters through should be thrown away.

The combined yield from the two crops of crystals is close to 50 grams. This packs the explosive punch of a comparable amount of nitro. For long term storage, the crystals should be soaked with water to absorb any acids they might generate during storage. It can then be filtered before uce

USE OF NITROMANNITE

In many respects the use of nitromannite is similar to the use of nitroglycerin. It requires about the same amount of force to set it off, and it delivers about the same yield of explosive power when compacted to rock form. There is a large difference, however, caused by the fact that nitromannitol is a fluffy solid whereas nitro is a liquid. This fluffiness means that nitromannite is nowhere near as dense as nitro is. As was mentioned earlier, to get maximum detonation velocity, and therefore explosive power, the explosive must be as dense as possible. So the big problem with nitromannite is to get its density high enough to deliver its full explosive potential.

The most effective way to compact nitromannite is to melt the substance by packing it into a glass vessel which will be its ultimate container, and melt the crystals by setting the glass container in warm to fairly hot cooking oil. The crystals melt at about 110ø C. When they cool down again, they will freeze into a solid rock. The same effect can be seen by packing snow into a container, melting the snow, and refreezing. The ice formed will take up quite a bit less space than the packed snow, so the substance is then much more dense.

This method is not to be recommended, because of the great danger involved. Nitromannitol becomes very sensitive at elevated temperature, so the heat required to melt large amounts of nitromannite could precipitate an explosion. The Home Workshop experimenter will have to settle for the increase in density which can be obtained just by packing the crystals into place. Care must be taken to avoid friction during the packing process.

The serious experimenter may wish to try packing the crystals into a vessel, then wetting the crystals down by spraying them with some ether (starting fluid). This will melt them, and when the ether has evaporated from the container, they will be rocked and take up less space.

A different approach takes advantage of the properties of nitromannite to create a self-detonating booby trap device. In the methyl nitrate section, mention was made of an attack plan featuring a bottle of methyl nitrate taped with heat resistant tape or other loose yet durable attachment to the exhaust manifold of a car. Nitromannite is much more suitable for this usage for several reasons. First of all, it is safer to make. Secondly, the heat from the exhaust will first melt the crystals, making them much more dense and more powerful. Finally, nitromannite will not half boil away before it explodes like methyl nitrate will (boiling point of methyl nitrate, about 65øC). For these reasons, nitromannite is much more likely to give satisfactory results in this attack plan.

In a similar vein, attack plans utilizing fuses in contact with exhaust manifolds leading to detonators, and nitrogen triiodide crystal laid down to set off the nitromannite from the vibration of the engine cannot be ignored. A fertile imagination is not only a joy for life, but also the key to improvised detonation systems. Good results can be obtained by routes other than the standard blasting cap-explosive charge combo.

A pretty good plastique can be made from nitromannite, just by mixing it with vaseline. Other, more advanced plastiques can also be made with nitromannite by preparing silicone polymer gels and so on, but the marginal advantages of these plastiques (they can be used under water, and in very hot places) are hardly worth the greater difficulty of preparation they present. Also, the ingredients used to make these gel matrixes are not the type of things you go down to the hardware store to get. Unless you work in the plastics or coatings field, you are unlikely to have easy access to them in less than 5 gallon pail quantities.

To make the plastique with vaseline, just mix 95 grams of nitromannite with 12 grams of vaseline in a stainless steel bowl. They can be mixed together by use of a rubber spatula. When a uniform mixture is made, the plastique can be wrapped in wax paper until use. A number 5 blasting cap or equivalent will set it off.

PETN

PETN, or pentaerythritol tetranitrate, is considerably more difficult to make than the other explosives considered to this point. Its preparation is not recommended for the casual experimenter, or for those with a clumsy streak in them. Its power and sensitivity is comparable to the other explosives in this book. It is also a crystalline solid, so it offers the same difficulties as nitromannitol in getting it compacted to maximum density for maximum power. If it were not for an extremely powerful and versatile plastique which can be made by mixing PETN with nitroglycerin, it would not be covered in this book.

There are several new difficulties encountered in the manufacture of PETN which combine to make this process a good deal more difficult than the preceding ones. First and foremost of these obstacles is the starting material, pentaerythritol (pentaerythrite). While it may be commercially available at about $20 per pound, its purchase is definitely not advisable for the Home Workshop Explosives manufacturer. Its purchase would leave and too recognizable paper trail, and could easily lead to suspicion or investigation either before or after usage. This leaves as the only viable alternative, making the pentaerythritol from simpler materials which are not subject to scrutiny, a tactic well known to drug manufacturers. This necessity to first make pentaerythritol makes this process a two-stage affair, with all the problems inherent to this approach. The biggest difficulty, other than all the work involved, is that the pentaerythritol must be refined to a high degree of purity before it can then be fed into the final stage of the process for conversion to PETN. The old computer programming slogan "garbage in, garbage out" holds doubly true in synthetic chemistry.

Another bad point about the two-stage nature of this process is the great amount of time which is required to make and purify the pentaerythritol. The unavoidable consequence of this is that this part of the process becomes a bottleneck in the manufacturing operation. The amount of product which can be made in a given period of time will always be controlled by how fast the pentaerythritol can be turned out to be fed into the PETN stage of the process.

Finally, the quality of nitric acid used to make PETN is crucial. This process is pretty fussy in that white fuming nitric acid must be used. This means that it can have no trace of the reddish gas NO_2 in it. A little bit of it in the mixture will in short order lead to runaway reactions and the formation of large clouds of NO_2, the dreaded red gas. This ruins the batch, and poses the danger of poisoning the experimenter, and of possible explosion.

Now let's look more closely at the first stage of PETN manufacture, making pentaerythritol. This adventure in chemistry is custom made for those who like to handle large volumes of really revolting chemicals and do endless hours of labour to get a quantity of product measured in fractions of a pound. Look once again at the materials covered earlier in this book.

Pentaerythritol is made by reacting formaldehyde with acetaldehyde, condensing them together with the help of calcium hydroxide.

$$8\ CH_2O + 2\ CH_3CHO + Ca(OH)_2 \Rightarrow 2\ C(CH_2OH)_4 + Ca(COOH)_2$$

Formaldehyde + Acetaldehyde + Calcium Hydroxide => Pentaerythritol

There is not too much which can go wrong with this reaction, so long as reasonable care is taken to follow the directions. It is, however, very stinky and potentially unhealthful if the experimenter allows himself to breathe in the fumes of formaldehyde and acetaldehyde. This reaction is best done outside with a steady breeze, the "cooker" keeping himself upwind throughout the process. If the watchful eyes of neighbours preclude this, a garage with a strong wind flow is acceptable. This may obscure the view of civic-minded citizens, but their noses are another enemy to be remembered. This process is best done in a secluded area.

To begin production, a clean plastic 5-gallon pail is filled with 2160 grams (2000 ml) of 37% formaldehyde solution, 210 grams (275 ml) of acetaldehyde, and 4 quarts of water. These chemicals do not need to be of a particularly high grade, so if money can be saved by using technical grade chemicals instead of reagent grade, then do so. Also, the formaldehyde solution can be replaced by 800 grams of paraformaldehyde. This solid form of formaldehyde does not have the powerful smell of the formaldehyde solution, but is much more expensive than regular formaldehyde. The 37% formaldehyde solution may be sold under the name of formalin, so be aware of this example of the proliferation of chemical synonyms.

Next, a clean wooden stick must be obtained. A section of broom handle minus the finish is a good example of what is called for here. This wooden stick is used to stir the solution.

First mix the ingredients already in the pail, then begin adding powdered quicklime (CaO, calcium oxide) to the pail in small portions with vigorous stirring. When the calcium oxide goes into solution, it first picks up a molecule of water, becoming $Ca(OH)_2$, and then takes part in the reaction shown above. The CaO should be added at such a rate that the temperature of the mixture rises to 50ø C (a little over 120ø F) within the first half hour of adding the CaO. Then the CaO is continued to be added at such a rate that the temperature of the mixture does not go over 55ø C (about 130ø F). As can easily be imagined, the fumes of formaldehyde and acetaldehyde get pretty intense as the solution gets hot. They get less revolting as the reaction nears completion and the aldehydes get consumed. The total amount of CaO added is 180 grams.

When all the CaO has been added, the stirring is continued at a more leisurely pace for another three hours. This long stretch of stirring is bound to tire even the most dedicated explosive manufacturer so an alternative which can be used where electricity is available is to suspend an electric drill or similar motor over the pail, and use a clean paint stirrer attachment to stir the solution. The fumes are not especially flammable, so fires are not the hazard they often are when dealing with more flammable chemicals. Even so, rigging an extra long stem for the stirrer, so the motor is elevated above the pail rim, is a wise precaution.

When the stirring is done, it is time to filter the now yellow colored solution. A large coffee filter fitted inside a plastic funnel will do a good job of this. The total volume of liquid amounts to about 3 gallons. Contained in this 3 gallons, is about 3/4 pound of pentaerythritol. Now the real work begins as the workaholic explosives manufacturer isolates his product from the mixture.

First, the mixture must be made slightly acid. To do this, hydrochloric acid (the 28% strength material available from hardware stores is good enough) is diluted 50-50 with water. Then this diluted HCl is added to the mixture with stirring until the mixture is acid to litmus (turns blue litmus paper red). A good way to do this is to add 100 ml of the dilute HCl right away, and then after stirring and checking for acid reaction, add smaller amounts of acid until an acid condition is achieved. This will convert the calcium formate made in the reaction to formic acid and $CaCl_2$, and also knock out left over CaO. In these forms they are more easily gotten rid of.

Next, the yellow color can be removed by adding 30 grams of activated charcoal powder (Norite brand is usually used in the lab) and stirring it around for a few minutes. Then the solution is once again filtered so as to remove the charcoal, and the filtrate is clear once all the charcoal has been successfully filtered out. Until then it is black, and the pail is a holy mess. Get a clean pail. This step can be omitted, but a yellow product will result which will be more touchy to convert to PETN without the dreaded red gas being formed. It will also not keep so well.

Now the solution must be reduced in volume so that crystals of pentaerythritol can form. To do this the water and other assorted smelly gunk must be boiled away under a vacuum. Formic acid and the unreacted aldehydes will be eliminated in this process. The first step in this adventure is to get a large enough container to hold the reaction mixture for the boil down. 5-gallon flasks are not commonly available to the public, but a good substitute is one of those thick glass water jugs often seen in offices for the water cooler. This is about 5 gallons in volume, and has a narrow opening which can be plugged with a one hole rubber stopper, and attached to the vacuum source (either aspirator or water bed pump).

To get this process going, put the reaction mixture into the glass jug along with a couple small pieces of a Dr. Scholl's pumice footstone (to ensure an even boil) and a chunk of paraffin wax the size of a small grape (to control frothing). The jug should be heated by means of steam, which can be supplied from a pressure cooker by filling it half full of water, clamping a section of automotive hose to the outlet on the lid where the weighted pressure control usually sits, and piping the steam produced from heating the pressure cooker into a cowling surrounding the jug.

The jug should sit in a large pan and be lifted off the bottom an inch or so by use of a few wooden blocks. The steam hose is run under the jug so that the steam rises up around the jug to heat it. The cowling can be as simple as a plastic garbage bag draped around the jug. A drain hose should run from the bottom of the pan to a drain or sink to carry away the water formed from the condensing steam.

When the jug is reasonably warm, vacuum should be applied to it and the heating continued. The contents of the jug will begin to boil away. This should be continued until the volume of liquid in the jug is reduced from 3 gallons to one gallon. Then the heating is stopped, and when the boiling ceases, the vacuum is removed.

Now that the liquid has been concentrated, crystals of pentaerythritol can form. Just let the jug cool off in the refrigerator overnight. In the morning, the crystals can be filtered out.

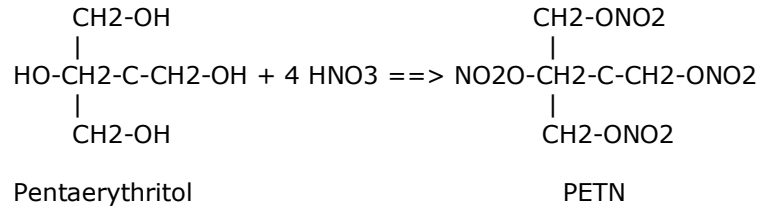
The liquid that filters through contains more product. This can be obtained by boiling away until the volume of the liquid is halved, i.e., reduced to 2 quarts. Upon cooling, a new crop of crystals can be filtered out. Repeating the process again, and boiling away the liquid down to one quart gives, upon cooling, another set of crystals. The remaining liquid can then be flushed down the toilet.

The crude product should be purified before use in PETN production. To do this, it is weighed, and an equal weight of distilled water is put into a stainless steel pan, or large pyrex beaker. The volume of water will be about a pint. The water is heated up on the stove, and the crystals are put into the water, along with 10 ml of hydrochloric acid. Mix them around until they dissolve, and boil just a little bit. Upon cooling, a large mass of crystals will appear. Filter them out. The liquid should then be concentrated down to about half its starting volume, and then cooled. Another crop of crystals will appear. By repeating this process a couple more times, about 350 grams of pure pentaerythritol will be obtained. It may be somewhat yellowish, but will work for making PETN. This product should be spread out on wax paper and allowed to dry thoroughly before use.

CONVERSION TO PETN

Compared to making pentaerythritol, PETN manufacture is a breeze. The amount of labour in the process is much less, and the volumes of chemicals which need to be handled are trivial in comparison.

The reaction here is the standard nitric ester reaction. In this case, pentaerythritol is the alcohol, and it is nitrated by means of nitric acid to form the nitric ester pentaerythritol tetranitrate (PETN). Here four molecules of nitric acid are needed for each molecule of pentaerythritol. See below:



Yields are very good, with one gram of pentaerythritol giving 2 grams of PETN.

To begin manufacture, the nitric acid is first checked. It must be fuming nitric acid (90%) and the acid must be completely water white. Any traces of yellowish or reddish pink color, and the stuff must be purified according to the directions below. The fuming nitric acid made according to the directions in the nitric acid section is usually water white, especially if the vacuum used was strong.

To decolor nitric acid, 800 ml of the fuming nitric acid is poured into a clean, dry 40 ounce beer bottle, and warmed up by setting the bottle in a sink of hot water. When it is warm, a gram or so of urea is added to the bottle and mixed in. Now dry air is blown through the acid. The most convenient way to do this is to plug the top of the beer bottle with a 2-hole stopper. Through one of the holes, a section of glass tubing is run. It should be long enough that it extends into the middle of the acid.

To get air flow, apply a vacuum to the other hole. This will pull air down through the glass tubing into the acid. Continue the air flow for a couple minutes and the color will be gone. Watch out for acid splashing up the tubing when the vacuum is removed.

Once the acid is clean enough to use, making PETN with it is fairly easy. First the 800 ml of nitric acid is poured into a large reaction vessel made of glass, with a pouring lip. A beer pitcher is a natural choice as the reaction vessel. The beer pitcher is nestled into a tub of ice. The ice should be heavily salted as in the nitromannite process in order to lower the temperature of the ice. The reaction temperature must be kept below 5ø C (about 41ø F) so salted ice is required to do this cooling job. As in the other processes, a pail of cold water should be close by to dump the batch into if it should go out of control.

Next, 200 grams of pentaerythritol is weighed out. It is then put onto several clean plates and ground up into a fine powder by crushing the crystals with the bottom of a drinking glass, or other handy object. Once they are ground up, the temperature of the acid should be checked to make sure it is below 5ø C. The temperature of salted ice can get down to 0ø F, so the acid should cool quickly.

When the temperature of the acid is low enough (stir the acid to get an even temperature throughout the pitcher) pentaerythritol can be added to the acid in small portions with good stirring. The temperature must be closely watched during the addition to make sure it does not go above 5ø C. Using the thermometer as the stirrer takes care of both problems at once. The reaction gives off heat, so the pentaerythritol must be added fairly slowly, especially if the acid was not very far below the 5ø C upper limit to begin with. When the upper limit is neared during addition of pentaerythritol, stop adding it, and continue stirring until the temperature falls enough to allow putting more pentaerythritol into the batch.

When all of the pentaerythritol (200 grams) has been added to the acid, continue stirring it in the ice bath for another 15 or 20 minutes. Acceptable colors for the reaction mixture are clear, white, and yellow. If its color is red or pink, be careful as an eruption of the dreaded red gas may be about to ensue. Be watchful for fumes bubbling from the batch.

At the end of the 15 to 20 minute stir period, it is time to end the reaction and begin to collect the product. To do so, the first step is to dump the batch into about a gallon and a half of ice water. Ice cubes should be floating around in the water because when the batch is dumped into the water, a good amount of heat will be given off. This is the heat of dilution of the nitric acid. A plastic pail with a pour spout is a good choice because after the batch is dumped into the ice water, it must be filtered, a job requiring it to then be poured out of the pail into a funnel.

So the batch is dumped into a gallon and a half of ice water. It is a good idea to stir the ice water as the batch is poured in to get the acid dispersed throughout the water. As the batch is dumped into the water, crystals of PETN will form. They will be white to yellow in color. They should be allowed to sit until the ice cubes melt in the pail.

Now it is time to filter out the crude crystals of PETN. A large coffee filter (cafeteria coffee maker size) put into a large plastic funnel will work well. Pour the batch into the funnel at whatever rate it can handle. The acid water that filters through can be thrown away. The crystals in the filter are rinsed by pouring cold water over them while still in the

funnel. About a quart of cold water, slowly poured, trying to reach all crystals will do the job. Some crystals will still be clinging to the sides of the pail into which the batch was dumped, so a good idea is to flush them out of the pail with some portions of water and add them to their brothers in the funnel. The best time to do this is just before the one quart cold water rinse.

Now the coffee filter is bundled into a ball and the water is squeezed but of it. After the water is squeezed from the crude crystals the traces of acid on the crystals are eliminated. To do this, a couple quarts of distilled water are mixed with 20 grams of Arm & Hammer~ bicarb and then the bicarb water is heated almost to boiling. This is poured into a clean beer pitcher, and the crude PETN crystals are mixed in. They are kept in contact with the bicarb water with some stirring for about an hour. It is not required that there be constant stirring. Just enough to keep the crystals floating around (once every 5 minutes or so will give good results) is all that is needed. Then the crystals are filtered again, rinsed over with some more distilled water (about a cup or two), and then squeezed out. These crystals are then spread out on some wax paper and allowed to dry before moving on to the final stage in the purification scheme, recrystallization.

Recrystallization must be done because of the traces of acid locked inside the crystals from when they were originally formed when the batch was dumped into the gallon and a half of ice water. All of the rinses have not been able to get at the insides of the individual crystals. Also, the yellowish crud which was tainting the pentaerythritol will be removed during the same process, resulting in a fairly white PETN. The purer PETN keeps better and stores more safely.

The solvent used to recrystallize PETN from is acetone. A good quality acetone can be purchased by the gallon at the local hardware store. Look in the paint section. PETN's very soluble in acetone, so it is not the ideal recrystallization solvent, but it is common and cheap. Besides, the suggested method found in the scientific books can be modified to give better results.

To start the recrystallization, the crude crystals of PETN, amounting to around a pound of product, are put into a glass container of about a quart capacity. An equal weight (about 600 ml) of acetone is added to the crystals of PETN, and they are mixed with a glass or wooden rod. Do not use plastic as the acetone dissolves most plastics. A great deal of the PETN will quickly dissolve into the acetone.

Next, the acetone must be heated to dissolve the rest of the PETN. Since acetone is very flammable, great care must be used during the heating to avoid a fire. This operation should be done in a garage or other structure that is open to wind blowing through it. Good ventilation is a must! Smoking is an invitation to disaster! Direct heat must also not be used to warm up the acetone. For example, setting the container holding the acetone/PETN mixture on a hotplate is a no-no because the fumes of acetone will come into contact with the burner and ignite Likewise, a double boiler set-up is not going to work.

The acetone can be heated by boiling a large pan of water in another room, and carrying the boiling water to the garage and placing the container into the hot water. As an alternative, a pressure cooker half-filled with water can be set onto a hotplate upwind from the acetone, and steam from the pressure cooker can be carried via hose to the acetone solution, just as was shown in the drawing in the pentaerythritol section of this chapter.

When the acetone gets hot, the remainder of the PETN should dissolve with some stirring. If it fails to all dissolve, add some more acetone. The acetone should be heated to boiling (56ø C) for maximum dissolving power.

Once all the PETN dissolves into the acetone, the first crop of pure PETN can be obtained. To get this first crop of crystals, just let the acetone solution cool down, first by removing it from the heat, then by packing the container in ice, and finally by putting it in the freezer. As the acetone gets colder, its ability to dissolve PETN decreases, and crystals of PETN form.

These crystals of PETN are filtered out either by using a vacuum system as shown in the nitromannite section (best) or by pouring them through a coffee filter and wringing them out. Plastic funnels must be avoided, of course.

The acetone which filters through still contains a very large amount of PETN. It is returned to the quart-size glass container and half the acetone is boiled off. This is best done by means of steam directed at the bottom of the glass container, via a hose from a pressure cooker. Acetone boils away quickly and easily.

When half of the acetone has boiled away, the heat is removed, and once again the acetone solution is cooled down. This results in a second crop of crystals. They are filtered out, and the acetone which filters through is returned to the glass container.

This acetone which remains still contains a lot of PETN. The best way to get the PETN out of the acetone which remains is to drip 190 proof grain alcohol (vodka) or 91% isopropyl alcohol into the acetone with stirring. As alcohol is added to acetone, its ability to dissolve PETN decreases. This is because PETN doesn't dissolve well in alcohol, so adding it to the acetone makes the solution lousy at dissolving PETN. This method is much better than boiling away more acetone because that method leads to a final crop of crystals that are dirty. When the solution is about half alcohol, all the PETN should be out of solution in the form of crystals. This is filtered out, and the acetone-alcohol mix is then tested to see if all the PETN is out of it by adding some more alcohol to see if more crystals form. If they do, alcohol is added until no more crystals form.

If the PETN is coming out of solution in the form of an oil that sinks to the bottom of the container, this indicates that it is pretty impure, and that the alcohol was added too fast without enough stirring. All that can be done in these cases is to put it in the freezer, and wait for it to solidify into crystals.

The PETN crystals are transferred to a large sheet of waxed paper, spread out and allowed to dry. When the solvents have evaporated from them, the PETN can be processed into plastique. My favorite PETN plastique is made by mixing PETN with nitroglycerin. I like it because it is extremely powerful (more powerful than C-4), easily detonated, and made from readily available materials. Its lone drawback is that the ease of detonation also means that rough handling could set it off. It must be treated with respect.

To make this plastique, 80 grams of PETN are put into a stainless steel bowl, and then 20 grams (12ml) of nitroglycerin is poured over the crystals. This mass is mixed with a rubber spatula until an even, uniform plastic mass resembling PlayDough is made. This substance can be wrapped in wax paper until used. A large firecracker or cherry bomb will set it off. Nitroglycol can be used in place of nitroglycerin to give a material which works at low temperatures and which is somewhat harder to detonate.

Another plastique can be made from PETN by mixing 98 grams of PETN with 12 grams of vaseline in the same manner as with the previous plastique. This forms a plastique which is almost as powerful as C-4 and is able to withstand rough treatment like C-4. A number 6 blasting cap is required to set this plastique off. Of course, a sufficiently large piece of nitrogen triiodide will also work. This plastique has the disadvantages of not tolerating high temperatures well (the vaseline starts to melt and run away) and also not being usable underwater since the plastique will disperse into moving water

Other plastiques can also be made from PETN, and they can be made to very closely resemble C-4, but their manufacture requires the use of materials which are not readily available to the typical consumer. Since ordering these materials would leave a paper trail leading to the workshop, it is not recommended to try to duplicate C-4. The very minimal advantages are far outweighed by these other, more pressing considerations.

RDX

RDX, also called cyclonite or cyclotrimethylenetrinitramine, is the last of our fine family of easily prepared, high power explosives.

In many ways RDX bears a striking resemblance to PETN. It is about as sensitive and powerful as PETN, and both are crystalline solids which can be made into plastiques. Beyond this, the manufacturing processes for these materials are quite similar as well. In both instances, a two-stage manufacturing process is called for because the starting material for making RDX may be difficult to obtain without causing suspicion.

The starting material for making RDX is a substance called hexamethylenetetramine. It may also be sold under such names as methenamine or urotropine. If it can be quickly purchased on a cash basis, a price of about $15 per pound is reasonable. If, on the other hand, this substance can't be so easily obtained, then once again, the route to follow is to make the starting material from simpler, non-suspicion-arousing materials. Luckily, hexamethylenetetramine is much quicker and easier to make than pentaerythritol.

There is another completely different method for making RDX, and it does not make use of hexamethylenetetramine as the starting material. However, that process requires acetic anhydride as a key ingredient. Acetic anhydride is central to the manufacture of several drugs, for example heroin and phenylacetone (the starting material for methamphetamine). For this reason, acetic anhydride purchases, and that whole other method of RDX production are to be avoided.

One could conceivably produce the acetic anhydride from simpler materials and then feed that material into RDX manufacture, but that is not such a good idea. The reason for this is because acetic anhydride manufacture centers around first making a substance called ketene (not ketane as was written in a pretender publication). This ketene stuff is god awfully nasty fearfully poisonous, and to make things worse, is a gas. Its preparation is something which should only be attempted by a real pro with proper equipment. Its preparation is nowhere near so simple as would be inferred from reading those pretender publications. To get an idea of what is really called for in ketene production, check Organic Syntheses, collective volume 1, pages 330 to 334. Also, using the ketene to make acetic anhydride is not so simple because an extremely effective fractionating column, almost a yard long, is required to get a clean product.

The method given here is much better because it uses simple and cheap ingredients that are easy to obtain. They are also pretty safe as far as chemicals go, with the exception of the fuming nitric acid. Also, these reactions are fairly easy, and result in mixtures that are pretty easily purified.

A well-ventilated garage is a good site for making the hexamethylenetetramine. The neighbours should not pay this process so much attention as the pentaerythritol process because the odour here is less intense and much shorter-lived. No special equipment is needed for this reaction either. All that is required is an electric hotplate with variable heat control, and a large enamelled dish to do the reaction in.

This large enamelled dish should be at least a gallon or so in capacity, and built to be able to withstand some heating and rough treatment. A very good choice is one of those roasting pans built to hold turkeys or hams. Look for one that is square or rectangular in shape so that the liquid may be poured out of the pan at the corner. The enamel can be substituted for with Teflon coating.

To make hexamethylenetetramine, 2000 grams (1850 ml) of 37% formaldehyde solution is poured into the enamelled dish. Then to this is added 1100 grams (1200 ml) of ammonium hydroxide solution. As was mentioned before, this material is just ammonia dissolved in water, and may be sold under such monikers as strong ammonia solution 27%, or 58% NH_4OH or ammonia water 26 degree balme. The ammonia solution should either be cooled down in the freezer before use, or have 250 grams of ice added to it.

The ammonia is added slowly to the formaldehyde with good stirring. A fair amount of heat is given off in the reaction, and the reek of formaldehyde and ammonia will get intense for a while until all the ammonia has been added. Then all the formaldehyde will be tied up, and only a weak smell of ammonia will remain. Stir some more to be certain that an even solution has been obtained.

The solution in the pan or dish contains about a pound and a quarter of product dissolved in a bunch of water. The task now is to obtain the hexamethylenetetramine, which is a crystalline solid, from the water. To do this, first the dish is placed on the hotplate, then heat is applied to it to bring it to a slow boil. As the water boils away, portions of about 15 ml of the ammonia solution are added to it every ten minutes or so. This is to ensure that enough ammonia is present to keep any formaldehyde which may be floating around unreacted, tied up.

When about half of the solution is boiled away, the heat should be removed from the pan and the solution is allowed to cool down to the point where it is about as hot as hot tap water. Then it is filtered through a coffee filter placed inside a plastic funnel. This is to remove any gummy material which may have been formed from the formaldehyde. This gummy stuff will quickly plug the filter, so be prepared to change the filter several times.

The liquid which filters through still contains the product. It is returned to the pan. Its volume should be a little over 2000 ml. It is once again boiled away until its volume is around 800 ml (1/3 or so of the previous volume). Now the

first crop of product can be obtained. Remove the pan from the heat and allow it to cool, then cool it further either by packing the pan in ice or putting it in the freezer. As it cools, a large mass of crystals will appear.

These crystals are then filtered out by filtering them through the vacuum filtering apparatus shown in the nitromannite chapter. Feel free to pack them down a little with a spoon to help squeeze the liquid out of them. Then the crystals are dumped out onto wax paper. The liquid should go into a separate jug for temporary storage until all the crystals are cleaned out of the pan. A rubber spatula will be very handy for scraping the cling-one off the walls of the pan.

When the first crop has been collected, the liquid is returned to the pan. Then the crystals of product are rinsed off. To do this, they are packed back into the filtering funnel, and rinsed off with 190 proof grain alcohol. A few rinses should remove the smell of ammonia from them. Then they should be spread out on fresh wax paper until they are dry. They should have no smell. They should also be clear or white in color.

The liquid which has been returned to the pan still contains a lot of product. To get it, first pour the liquid into a glass beer pitcher or similar container. Then about 10 grams of powdered activated charcoal is added to the liquid. If the lab brand of charcoal called Norite is available, this is best. Otherwise, the various other types of activated charcoal will work so long as they are powdered up before use.

This activated charcoal will take up the yellowish color from the solution. First stir it in for a few minutes. Then set up the vacuum filtration system again, with double layers of filter paper. Pour the solution through it. A clear solution should result. If it is still black, all the charcoal isn't out of it. Wash up the system, change the filters, and filter some more. If it is still yellowish, add some more activated charcoal and continue.

The clear solution can now be returned to the original dish, and heat reapplied to it. The goal here is to evaporate the liquid to dryness and collect the crystals which are left when the water is gone. Heat it reasonably strongly at first, then steadily back off on the heat as the volume of liquid goes down. One does not want to burn the crystals. Finally, end up with just a warm setting, and drive off the last of the water. Then scrape out the crystals of product from the pan. The total yield of hexamethylenetetramine should add up to around 560 grams.

All the hexamethylenetetramine should be spread out onto wax paper sheets and allowed to dry. When it is thoroughly dry, it is ready to be turned into RDX. Very humid weather may cause the crystals to soak up water from the air and melt, so beware of spreading them out to melt instead of dry.

PREPARATION OF RDX

Once the hexamethylenetetramine is dry, one is ready to proceed to turn it into RDX. This reaction is a good deal more touchy than the ones covered in the earlier sections of this book. If directions are not followed concerning acid strengths, temperatures, and reaction times and conditions, the dreaded red gas is likely to appear, or at least the yield of product will be ruined. Have no fear, however; all the little things which are likely to go wrong will be covered, along with the several right ways to do this reaction. So long as one stays on the right path, and off the road leading to disaster, all will be well.

RDX, unlike the other explosives covered in this book, is not a nitric ester. This means that the equilibrium equation presented back in the early part of the book can be ignored for this explosive. Instead, the factors most influencing yields in this reaction are kinetic. This means that there are several side reactions which occur at the same time as the desired reaction, and getting a good yield depends upon suppressing these side reactions, and encouraging the desired reaction.

One encourages and suppresses reactions by carefully controlling the time of reaction, the temperature(s) at which it is run, and the concentration of the ingredients, among other ways. Just how this strategy works for RDX production must now be explained.

First, we have the desired reaction, producing RDX from hexamethylenetetramine and nitric acid:

A very important competing reaction is the breakdown of the hexamethylenetetramine by water into its hydrolysis products, formaldehyde and ammonia:

$$C_6H_{12}N_4 + 6\ H_2O \xrightarrow[\text{Catalyst}]{\text{Acid}} 6\ \text{Formaldehyde} + 4\ \text{Ammonia}$$

The water finds its way into the mixture because water is the diluent for nitric acid, and even the 90% fuming acid obtained commercially is going to have 10% water as the diluent. The nitric acid made as directed in the nitric acid section will have next to no water in it, so long as the saltpeter was dry, and the glassware was dry. This is anhydrous nitric acid, and so avoids this side reaction, so long as the hexamethylenetetramine is dry, and the glassware is dry.

This side reaction is also greatly slowed down if the batch is made at a low temperature. The cold temperature slows down the unwanted reaction quite a bit more than the desired reaction producing RDX. By doing the reaction at -20ø C (0ø F), an additional one sixth of product is obtained. This side reaction is further curtailed if the batch is not allowed to sit around any longer than is absolutely necessary before pouring it into water and ending the reaction.

The best waiting period after the end of adding the hexamethylenetetramine to nitric acid, before dumping the batch into water, is about 15 minutes. This is about the length of time required for the last of the hexamethylenetetramine to dissolve into the nitric acid, with slow stirring.

Another side reaction involves nitric acid adding to the hexamethylenetetramine to form a salt. This side reaction is not so easily controlled as the preceding hydrolysis reaction. It just has to be lived with, and is the reason that the best yield one can get is around 75% of the theoretical yield.

With a good grounding in the ins and outs of this reaction, let's go on to see how these principles are put into practice Using the commercially available fuming nitric acid of 90% strength (density 1.51), the same set-up is used as for the previous reactions. A beer pitcher makes a good reaction vessel, and it should be nestled in a tub of ice, with a 5 gallon pail of water nearby to dump the batch into in case it goes out of control and begins spouting red gas. One must be especially on guard when doing this reaction, because the onset of the dreaded red gas can be the result

Then into the pitcher, 770 ml of the fuming nitric acid is placed. This acid should be cooled down beforehand by storing the acid in the freezer. Then 100 grams of hexamethylenetetramine is weighed out, and it is added to the acid slowly, in small portions, with slow stirring. A good tool to do this stirring is a section of glass rod, bent at a right angle about an inch before its end. This stirrer will look like an overly long hockey stick, and works like an agitator when slowly twirled between the fingers. Overly fast stirring is to be avoided, because this contributes to an overly fast reaction which may get out of control and start to fume. The temperature of the mixture should under no circumstances be allowed to go above 20ø C, and it would be best if it were held well below that temperature. Since one is starting with acid from the freezer, where its temperature should be around -20ø C, this should be no hard task The amount of time required to add all the hexamethylenetetramine to the acid is going to be about half an hour.

Then the slow stirring is continued, while a close watch is kept on the crystals of hexamethylenetetramine in the acid. When the last of them has dissolved, wait 5 minutes, then pour the whole batch into a gallon of cold water. The length of time between the end of adding the hexamethylenetetramine, and the point when it has all dissolved, is around 15 minutes. As was stated earlier, it is bad idea to let the batch sit around any longer than the recommended period. Once it has all dissolved, wait a few minutes, then pour it into the gallon of water.

When the batch is poured into water, crude crystals of RDX will form. They must next be filtered out, so for this reason, it is wise to choose a water container which will easily pour into a funnel for the filtering process. Three beer pitchers, each holding an equal volume of cold water ready to accept 1/3 of the batch, is a good arrangement.

Next the crude crystals of RDX are filtered out of the water by using the vacuum filtration set-up described in the nitromannite chapter. The best filter to place in the funnel is a piece of glass wool. This material can be found at any place selling aquarium supplies. It is called angel's hair. A piece of it is pushed down into place over the filter holes, and then wetted into place with water before beginning the filtration. This can be used for the nitromannite filtration as well.

Unlike the earlier explosives, the crude RDX can be kept and used as is, once it has been well washed down with water while in the funnel. Some litmus paper comes in handy to tell when the last of the acid is being rinsed from the RDX. If a drop or two of the rinse water doesn't turn blue litmus paper red, then the acid is rinsed off the crystals. If this crude RDX is stored for any length of time, it should be stored underwater.

This crude RDX can be purified very well by recrystallizing it from acetone. For every gram of RDX, 10 grams of acetone should be used. Ten grams of acetone is 12.7 ml. The recrystallization is done like the previous recrystallization from acetone. The acetone-RDX mixture is heated by setting the container in hot water, and the mixture is stirred until the RDX dissolves. Then the solution is cooled down by packing the container in ice. A large amount of really nice looking RDX crystals form. They are filtered out, and the acetone solution is then boiled down to half its previous volume. Upon cooling, another crop of crystals form. Finally, water is added to the acetone solution to get the last of the RDX to precipitate out. This RDX has no acid in it, and stores really well.

The yield of RDX by this method is about 60%, which translates to about 95 grams of RDX per batch. The yield can be increased by a few grams if double the stated amount of nitric acid is used, but this is bad strategy considering the cost of fuming nitric acid. A much better strategy for increasing yields is to use the anhydrous fuming nitric acid that can be made according to the directions given in the nitric acid section Use of this material gives a yield of around 110 grams per batch.

To use the anhydrous nitric acid, one sets out pretty much as in the previous example, except that the ice must be heavily salted. This is because the reaction must be done at a much lower temperature. A temperature of around -20ø C (0ø F) is needed to use the anhydrous nitric acid. If the temperature of the batch goes much above this, the familiar penalty of the dreaded red gas is promptly dished out. Dry ice soaked down with rubbing alcohol or acetone would do a great job of keeping the batch cold, if the dry ice is easily available in your workshop. Naturally, the nitric acid is taken straight from the freezer, and not allowed to warm up.

The same quantities of ingredients are used in this method as in the previous one. A big difference, however, comes in the length of time required to add the hexamethylenetetramine to the acid. This time period should be roughly tripled to 90 minutes, so that the heat given off from the reaction doesn't get too intense. Then the stirring is continued slowly until all of the crystals are dissolved. After waiting a further 5 minutes, the batch is dumped into water as before and the previous instructions can be followed the rest of the way.

When contemplating RDX production, one may be tempted to try to use the cheaper 70% strength nitric acid (density 1.42) instead of fuming nitric acid. This just does not work. At the usual temperatures, no reaction results, and at higher temperatures, red gas is made. There is an alternative method starting with 70% nitric acid, which is then watered down further, and hexamethylenetetramine is added to this. The resulting product is the nitrate salt which was briefly mentioned earlier as an unavoidable side reaction. This salt can then be isolated and added to fuming nitric acid to give RDX.

This route is not so good as the ones given above because more steps are involved, and the process is wasteful of hexamethylenetetramine. Also, the total yields are lower. Its only good point is that it uses roughly half as much fuming nitric acid. Since high strength nitric acid is easier to make than hexamethylenetetramine, the scales tip heavily towards the processes given here.

A very nice plastique can be made from RDX by mixing it with vaseline in the same proportions as for PETN. It is a little more diffcult to detonate than the PETN plastique. A number 8 blasting cap is called for in this case.

If the material is being used straight, it can be set off with a firecracker which has had some nitro soaked into the wrapping paper. To get maximum firepower out of the charge, it must be compacted to a higher density than the fluffy crystalline state. The only common solvent which works really well for melting the crystals into a rock is benzene. Good luck in finding that one at the hardware store! Acetone may do an acceptable job. Be sure to let the rerocked RDX set around until it no longer smells like acetone. The warmer the acetone, the better it will work for this purpose.

DETONATION SYSTEMS

Without a means of detonating an explosive, the products described in this book are nothing more than dangerous curiosity pieces. This often overlooked facet of explosive use generally is the point that trips up newcomers to the field. To procure or make explosive substances is a pretty straightforward operation. Rigging a detonation system, on the other hand, can seem mysterious by comparison. For this reason, conventional and improvised detonators must be covered. Since all explosives in this book are detonatable by these methods, reference to this section can be made throughout the book.

All explosives can be set off by either a sufficiently strong electrical shock, or by being struck a hard enough blow. The exact amount of power required by either of these methods depends upon the explosive in question. For instance, static electricity from walking across a carpet can be enough to set off guncotton, while TNT is notoriously hard to set off with a shock. The other explosives fall somewhere in between these two extremes. Similarly, TNT requires a heavy whack with a sledge hammer to set off, while nitro or gunpowder can be set off with a moderate blow from a hammer or similar metal object.

With this superficial intro to the topic behind us, let's move on to applying these principles to the practical task of detonating a Home Workshop Explosive. The simplest, safest and most reliable route to follow is if one has access to blasting caps. These little wonders are made specifically to do the job in question, and their performance is hard to top. So long as one uses the required size or larger, no problems will be encountered. The body of the blasting cap should be inserted into the explosive. With a large pile, a spot near the bottom is preferred.

If, on the other hand, one does not have blasting caps, then improvisation can lead to good results. For example, a good firecracker is sufficient to set off pure nitro. Delay mechanisms can be rigged by planting the firecracker fuse near the filter end of a cigarette, and then allowing the cigarette to burn down to the fuse. This will give a few minutes delay. Since many firecrackers are duds, it would be wise to rig several backups.

A #1 blasting cap equivalent can be improvised by putting into use the detonation train principle. This principle is often used to set off difficultly exploded substances like ammonium nitrate. Here it will be put into practice simply by dipping a good sized firecracker into nitro. The paper wrapping will absorb the nitro, and then the nitro will go off with the explosion of the firecracker, adding its punch to the blast. These nitro crackers are great fun by themselves. If you are bored by commercially available fireworks, they are highly recommended. The only pitfall in their manufacture is if the firecracker is wrapped in a sheet of waxy or glossy paper. Then this should be removed so that the nitro can soak into the coarser paper beneath.

Larger blasting caps can be improvised along the same lines by allowing the firecracker to set off a larger amount of nitro, or by packing a nitro-soaked firecracker into some gelatin, and allowing the ensuing detonation train to do its work.

Alternatively, a couple of good books list directions for making improvised blasting caps that more closely resemble the commercial product. The best of the lot are The Poor Man's James Bond and Kitchen Improvised Blasting Caps. Have fun!

The electric shock method of detonation is one that is often ignored, but one that can prove very useful for bagging elusive game. One of my favourite electric detonators is a spark plug. The newer cars with the electronic ignition send a lot more juice to the spark plugs than the older models. This spark, particularly from the hotter spark plugs, will suffice to set off nitro and usually nitroglycol. The possibilities for this method are bounded only by the imagination.

In putting this detonation method into practice, a clean spark plug should be used so that the explosive is not going to be fouled up with carbon deposits. To test out the method, a couple drops can be deposited on the electrode of the spark plug, and after attaching the plug to the distributor, the engine can be cranked.

Gelatin is a tricky substance to detonate by this method. Too weak a shock will just set it on fire with no explosion likely to result. Experimentation is the only way to tell if the particular model in question produces enough shock to bring on detonation.

Other sources of electric shock provide intriguing possibilities along similar lines. Working out the details are bound to provide hours of pure enjoyment for the serious experimenter.

A final method of detonation that is supremely simple and suitable for rigging booby traps makes use of a curious substance called nitrogen triiodide. This very easily made substance has the strange property of being pretty stable when wet, and yet wildly explosive when dried down to the crystal form. The smallest touch will set it off in this dry state. It explodes powerfully enough to set off nitro, all gelatins, and if used liberally, even dynamite.

Nitrogen triiodide is often made by high school chemistry teachers as a class demonstration to impress the students. Considering the sorry state of secondary education in this country, you can get an idea of just how easy this stuff is to make. If you are not brain damaged, you will have no problems.

To make nitrogen triiodide (NI_3) one first gets iodine crystals. These can be ordered without suspicion from any of the mail order supply outfits, or ordered from a pharmacy. If one is forced to deal with a pharmacy, these terminally snoopy people may inquire what they are needed for. Good answers to such nosey questions are that one is

experimenting with treatments for fish tank infections, or that you are stocking up on it so iodine tincture can be made in your bunker after they drop the big ones.

Iodine crystals (I_2) are black and heavy. A quarter pound should cost around $20. If much more than this is charged, you are being ripped off. The crystals should be spread out on a plate, and ground finely using the bottom of a small glass. It is very important that a fine grind is obtained, or the product is likely to go off by itself during the drying process.

The other required ingredient is ammonium hydroxide. This is just a strong solution of ammonia in water, and it can also be obtained with ease from mail order suppliers, or a pharmacy. The product one needs here is called ammonium hydroxide (NH_4OH). The strength level should be about 58% NH_4OH or 28% NH_3 or 26 degrees balme. All these mean the same thing; just one of the ways that chemists have to confuse the general public. Cost: no more than $10 per pint.

A product that will just not do is the ammonia which can be picked up in the grocery store. This stuff is just 3% ammonia, and is loaded with soap to boot. It can't be made to work, either, by distilling the ammonia out of it. The soap in it begins foaming like crazy as it is heated up, making this operation futile.

With the supplies on hand and ready, NI_3 is made just by mixing one volume of iodine crystals with about ten volumes of NH_4OH. A glass measuring cup is perfect for this reaction. They should be allowed to react for ten or 15 minutes with occasional stirring. Beware of breathing in ammonia fumes from the ammonium hydroxide during this process. Good ventilation is a must.

Then a drip type coffee filter is placed in a funnel or other suitable holder. The funnel is then placed in the mouth of a beer pitcher, or other suitable container to catch the ammonia water which will filter through.

Then the batch is stirred up well to suspend all the crystals in the liquid, and quickly poured into the filter. Any crystals sticking to the cup can be rinsed out with some more ammonium hydroxide, or scraped out with a rubber spatula.

The crystals of NI_3, which are black, should be lightly squeezed in the filter to free them from excess solvent. Then the paste-like product should be loaded, while still wet and stinking hke ammonia, into the corner of a zip lock baggie. Here they may be stored for a while in the refrigerator to await further experimentation.

To get the feel of this substance, a few lumps should be taken out of the baggie and spread out to dry on a tabletop or cement floor. When it is dry, the smallest touch with any stick or similar probe will set it off. The blast which results can be really surprising. Another good line of experimentation is to stretch the stuff out in a long thin line and see how thin it can be spread and still get the whole line to blow off when any part of it is touched. Also, you should practice to see how long it takes to dry out larger lumps of the material.

With this experimentation successfully finished, one can move on to the nitty gritty of rigging a booby trap surprise. A lump of NI_3 a tattle bigger than a pea will set off nitro with ease. A lump about twice that size placed against the outside of a bottle containing nitro will be sure of setting it off counting the shielding effect of the glass. A somewhat larger lump will detonate gelatin, and a little more will do the job with more difficultly exploded substances.

Since the explosive container is likely to arouse suspicion, it may often be good practice to run a thin line of the NI_3 from an area that the target will step on to the explosive stash. So long as the blast occurs close enough to the target, all will be well.

On the other hand, in many cases it is better to rely on the curiosity of the mark, and just set the lump against the bottle or baggie of explosive. When he comes to examine it, the small nudge will set off the NI_3 and result in detonation. Here ingenious disguises of the explosive booby trap can pay handsome dividends.

As should be obvious from the preceding sections, the NI_3 must be put into place while still wet, and have time to dry in place before it becomes activated for use. This is the reason why attention must be paid to drying times for various size lumps of NI_3. The time will vary with temperature and humidity because water is the main ingredient to evaporate off from the NI_3. NI_3 can be carried around safely in a baggie so long as it is still wet from being made. Once it dries, it is set to explode!

WARNING! The filter used in this process will itself become explosive when it dries because of the small pieces of NI_3 caught in it It should either be flushed down the toilet immediately, or saved and treated like the dangerous explosive it is.

APPENDIX 1: SAFETY

The acids used in the manufacture of these explosive compounds are dangerous and caustic materials, so a small bit of preaching is called for here to help prevent needless accidents from befalling careless experimenters. We all like to imagine that nothing bad can happen to us, but all it takes is a moment of inattention to result in a lifetime of misery.

The most crucial area of the body to protect when working with chemicals is the eyes. In comparison to the eyes, damage done to other parts of the body is minor and short-lived. For this reason, the sensible experimenter will wear clear plastic goggles. The best goggles are sold in college bookstores for use by chemistry students in their lab courses. These are cheap, easily bought and perfect for the job. An OK alternative is the large plastic eyeglasses with side shielding often used with chainsaws.

Eye protection should be worn whenever acids are being measured out, and while batches are being made. Not until the acids are washed away by flushing the product with water can the goggles be removed.

Some of the reactions in this book call for the use of chemicals like formaldehyde or acetaldehyde or ammonia. The same rules must be followed for these materials. They will cause damage if splashed into the eyes. Beyond that, their fumes can cause similar damage. Good ventilation must be provided for their use.

The acids used to make explosives can also cause serious skin burns if they are allowed to sit on the skin for more than a minute or so. Vigilance is the best protection against this hazard, along with a willingness to rinse one's hands regularly when working with the acids. Rubber gloves can also be worn to protect the skin. Surgical gloves are the best because they leave the experimenter with a degree of nimbleness in the fingers. Next best are the "living gloves" popular with dishwashers fearful of "dishpan hands."

When fuming acids are being used, gloves are a must. This is because the acid fumes will settle on exposed skin and begin to burn. The arms should be similarly protected in these cases by wearing long-sleeved shirts. The importance of good ventilation, and of keeping upwind of the air flow is obvious here.

If the unthinkable should happen, and large volumes of acids are splashed upon your body, time is of the essence. You must run directly to the shower, jump in with all your clothes on, and flood the affected area with cool or lukewarm water while peeling your clothes off. A good rinsing for 10 minutes or so is called for, and can be followed with a rinse of bicarb solution.

At the end of a day's work, or just while on break, you can bet there are droplets of acid on your skin you didn't notice. These can be wiped out with a leisurely shower. The clothes should go directly to the wash, or they are going to have holes eaten in them.

Glassware coated with residues of acids or explosives are a very real, but often overlooked, source of danger. All utensils used in these processes should be thoroughly rinsed with water as soon as possible after use. When the items in question have been in contact with substances that don't dissolve in water, they should be further rinsed down with rubbing alcohol or acetone until clean. When explosives are in solution, they are usually very hard to detonate. We can then consider these rinse materials safe to flush away. They may reprecipitate in the lines but a good follow up with bleach should destroy most of them.

Major spills of explosives should never happen to the careful and conscientious manufacturer. Minor spills can be dealt with by following some general guidelines. If it was batch material before drowning in water, dilute it with some water, then soak it up in a bunch of Kleenex. Follow this with some alcohol or acetone, depending upon what solvent the explosive dissolves in, and mop it up with some more Kleenex.

If it is more refined material, dilution with alcohol or acetone and mopping up with Kleenex is the first step. Then for the solid explosives, rub in some vaseline on the area, and continue wiping it up. If you don't get it all, at least it will be made more shock resistant. In all cases, the dilution and mop up should be continued until it looks like all of the explosive is gone The Kleenexes can then either be flushed away, or carefully bummed outside. An explosion may result from this latter course, so be prepared for the fireworks.

APPENDIX II: ASPIRATORS AND WATER BED PUMPS

The use of vacuum to filter liquids and to distill materials at more reasonable temperatures is central to many of the processes in this book. With the great importance assumed by a reliable source of vacuum for these operations, it would well serve any explosive manufacturer to become familiar with convenient sources of vacuum, and the hidden problems they pose.

The best source of vacuum for the lab operations described in this book is the aspirator, also called the water pump. Its bastard cousin, the water bed drain pump works the same way, but produces a weaker vacuum due to inferior engineering and construction. Let's take a quick look at these two devices to see how their performance can be optimized.

These devices work by running a cold stream of water through them. The rush of water down the body of the aspirator produces a vacuum in the side arm portion of the device. An automotive vacuum hose of the appropriate size can be attached to the side arm and led to the glassware needing vacuum. This hose should not be more than 3 feet long for highest strength vacuum.

Most aspirators and water bed pumps have a threaded section at their top to allow them to be screwed onto the end of matching threaded faucets. If this is not convenient or possible, then a section of hose or plastic tubing can be fitted to connect the aspirator and faucet. A couple of hose clamps to keep everything together are called for when doing this. The water exit of the aspirator must also have hose connected to it to lead to the drain. This cuts down on splashing from the stream of water, and also results in a better vacuum. The flow of water out of the aspirator should be unimpeded, and the exit hose should be of larger diameter than the input feed line.

Water for the aspirator should be as cold as possible for best results. This is because the vapour pressure of water at the particular temperature of water being used is the best possible vacuum from an aspirator. Since cold water has a lower vapour pressure, the best vacuum will come with cold water.

A most annoying habit that these type of vacuum devices have is their propensity to puke water back into their vacuum lines. The root cause of this behaviour is usually traced back to variations in water pressure or flow rate. Things like a toilet flushing or a well pump kicking on and off are typical culprits.

The way this back puking works is that a pretty good vacuum will get built up in a piece of glassware, just about equal to the maximum the aspirator can deliver. Then the water flow slows down and the amount of vacuum being produced by the aspirator goes down. The next thing that happens is that the stronger vacuum in the glassware proceeds to pull water down the vacuum line from the aspirator to the glassware. When the product one wants is anhydrous nitric acid or glycerin or ethylene glycol, this is a disaster. Good aspirators have valves to make this a less routine occurrence.

There are a few steps one can take to make this puking a very rare happening. First of all, city water supplies tend to have the pressure variations ironed out of them. No well pumps kicking on and off. This is the ideal water system for aspirators. Just turn the water on strong at the start and leave it there.

Secondly, the aspirator should be at a lower level than the glassware it serves. Six inches to a foot is a convenient level. This positioning of the aspirator forces any water that pukes back into the line to crawl uphill to get to the glassware. The delay won here is often enough to get the situation to straighten itself out.

Finally, watchfulness can catch a problem before it develops into a disaster. Be aware of the problem' and it cannot catch you by surprise.

BOILING POINTS UNDER VACUUM FOR ETHYLENE GLYCOL AND GLYCERIN

Since the best way to get a great quality ethylene glycol or glycerin from crude commercial mixtures is to vacuum distill the mixture to get the pure product, a table of boiling points for these two substances at various strengths of vacuum will be given to make it simpler to spot the product fraction. A fair amount of practice at vacuum distillation is generally needed before one becomes a pro at it. See a college level organic chemistry lab text for a detailed discussion of vacuum distillation. Both ethylene glycol and glycerin are good substances to practice with because they are pretty safe materials.

Perfect vacuum = 0 mmHg Normal Pressure = 760 mmHg øC

mmHg (boiling points)	Ethylene Glycol	Glycerin
1	53 øC	125 øC
10	92 øC	167 øC
40	120 øC	198 øC
100	141 øC	220 øC
400	178 øC	263 øC
760	197 øC	290 øC

A well working aspirator running on cold water will give a vacuum of from 10 to 25 mmHg. Of course, the temperature at which the product will boil in any particular experiment depends upon the strength of the vacuum being produced by the aspirator on the given occasion. This can typically vary by about 10 mmHg. Very bad vacuums should be dealt with by looking for leaks at the ground glass joints (always use stopcock grease on these joints!). Other culprits can be collapsed vacuum hoses or ill-fitting rubber, glass or Teflon stoppers. Corks can never be used because they always leak. Sometimes, the aspirator will be installed upside down. The threaded end should always be up.